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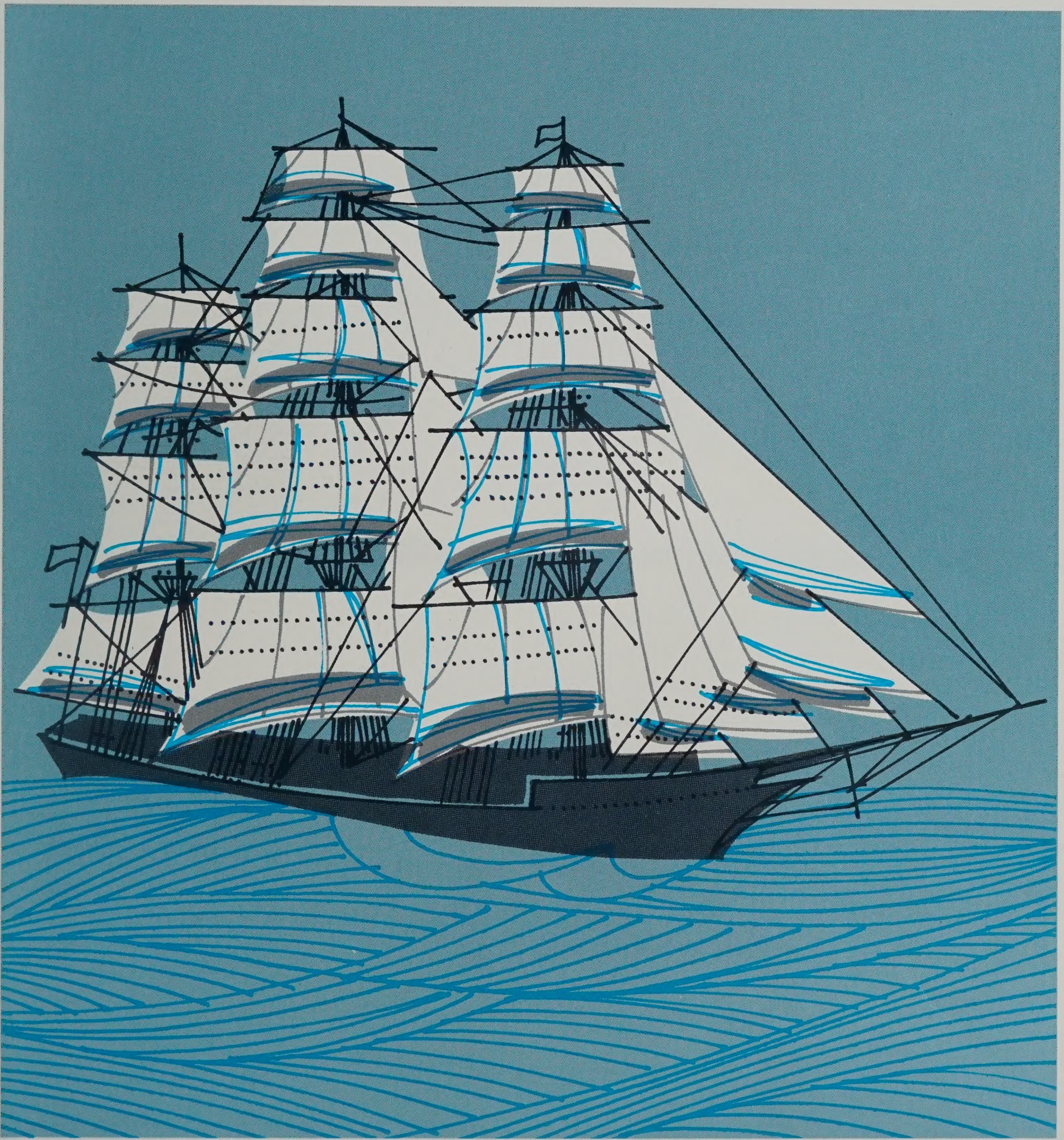

ANTIFOULING COATINGS FOR SHIPS. See page 12.

COMMENT

MEASURING THE CHEMICAL DURABILITY OF MATERIALS

This month's DIMENSIONS cover article, which describes NBS work in support of the U.S. Navy's development of improved anti-foulant coatings, illustrates at least three of the many themes in the Bureau's current work in the area of materials durability.

NBS has a long history of productive research on the chemical durability of materials. The Bureau's program in metallic corrosion, dating back to 1911, has provided data on materials performance as well as fundamental understanding of the science underlying the degradation of metals in both common and aggressive chemical environments. The national significance of this problem was underscored by the 1978 NBS study on the economic effects of corrosion, which showed that metallic corrosion alone cost the United States some $70 billion in 1975. NBS is currently expanding its corrosion research effort as part of an overall materials durability program in the Center for Materials Science.

One focus of current NBS work in chemical durability is research to develop measurement methods with which to probe at the molecular level the chemical reactions between materials and their service environments. In the case of the anti-foulant coatings described in this issue, chemical degradation is not all bad—it is, in fact, required for coatings to be effective. A "benign corrosion" process in sea water releases minute amounts of toxic chemical species containing tin atoms bonded to organic groups that inhibit the growth of marine organisms on the surface of the coating.

To support development of these coatings, new measurement methods were needed to measure the trace amounts of organotin materials released, and even more important to identify their specific chemical forms, which determine their biocidal activity. A completely analogous problem, also being studied in the Chemical Stability and Corrosion Division, is the measurement of chemical species formed when refractories or metals are attacked by hot, corrosive gases. The techniques used are very different, but the need for trace-level, compound-specific, *in-situ* measurements is the same.

Another theme apparent in the anti-foulant work is the need for measurement methods to evaluate and control the environmental impact of materials use. When a material corrodes or degrades, the degradation products enter the environment and are transported and transformed by chemical and biological action. The chemical durability of the degradation products, at the molecular level, ultimately determines their fate and their interaction with the environment. To track the products through their environmental transformations and determine their ultimate impact, the same sorts of trace-level, compound-specific measurements used to study the primary degradation process are required.

A third element in the chemical durability problem is the importance of cooperative action of chemical and biological processes. Microorganisms are efficient processors of materials. For example, microbial action can result in corrosion of iron or aluminum, accelerate the leaching of copper or other metals from minerals, or transform relatively innocuous inorganic forms of elements such as mercury into highly toxic organometallic compounds. These biological processes must be considered in any assessment of long-term materials durability. The measurement methods described in this issue are now being applied by NBS researchers to gain insight into microbiological processes that limit the service life of materials.

In order to predict and improve the performance and durability of materials in reactive chemical environments, it is essential to understand the nature of the chemical and biological degradation processes they undergo. The goals of NBS research in chemical stability and corrosion science are to develop the tools required for reliable measurement of critical species, processes, and rates and to apply those measurements to provide that understanding.

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Contents

ARTICLES

2 **Signs of the Times**
Measuring the Success with Which Signs Communicate

7 **Measurement and Industrial Development in the 1980's**
Many Challenging Issues Face the Measurement Community

12 **The End of the Free Ride**
Making Antifouling Coatings that Last Longer

Special
18 Improved X-Ray and γ-Ray Measurements Have Several Applications
γ-Ray Secondary Standards Bettered by a Factor of Forty

INTERFACE

25 **ON LINE WITH INDUSTRY**
NBS Opens New Industrial Furnace Facility

26 **STAFF REPORTS**
Surface Magnetism Studied Using Polarized Electrons
Definitive Measurement of Constituents in Human Serum

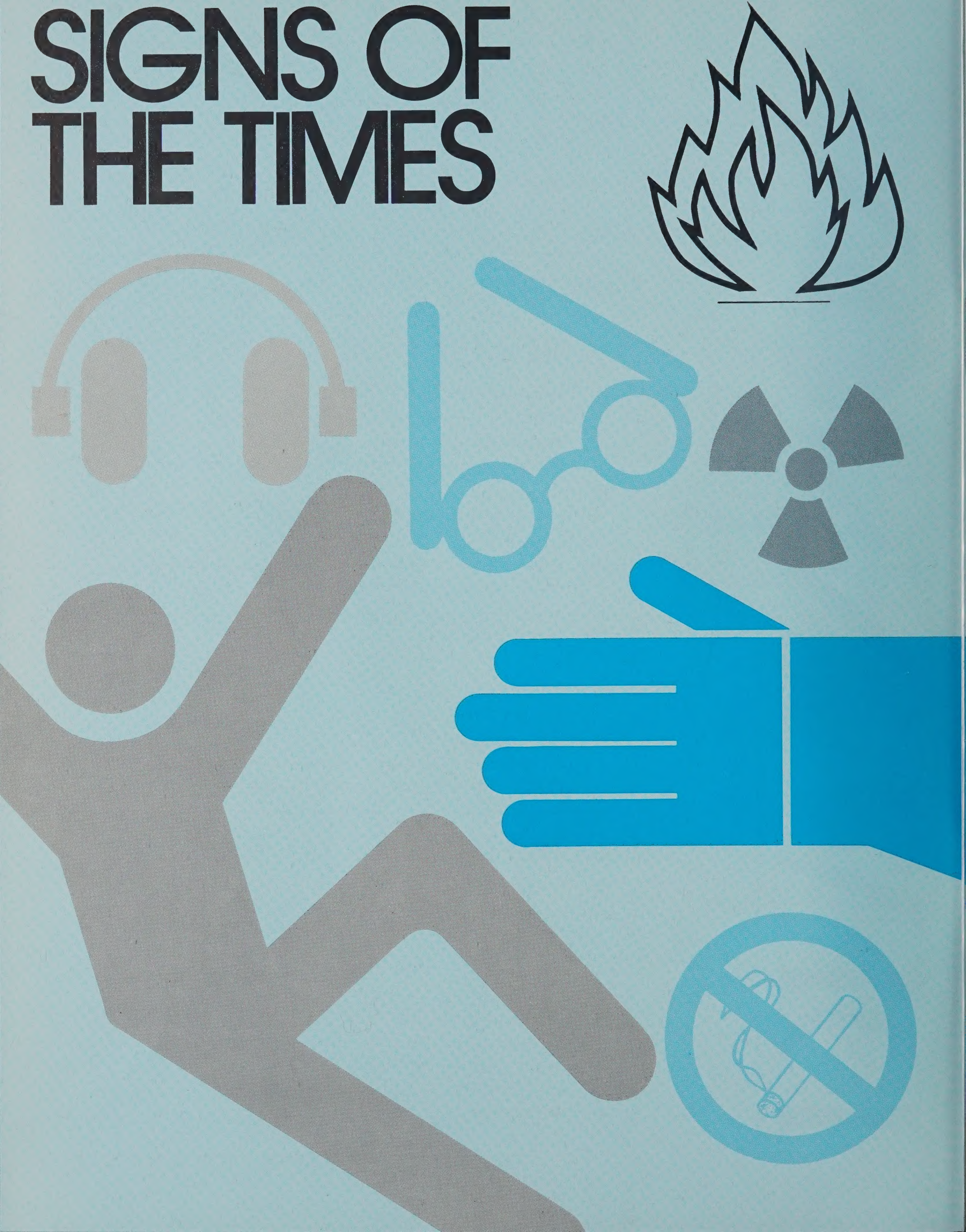
UPDATE

32 **CONFERENCES**
Implant Retrieval: Material and Biological Analysis Conference
Fifth International Symposium on Ultrasonic Imaging and Tissue Characterization
Second International Symposium on Ultrasonic Materials Characterization
Conference Calendar

34 **PUBLICATIONS**
Proceedings on the Characterization of High Temperature Vapors and Gases
Neutron Radiation from Medical Accelerators
NBS Publications

36 **NEWS BRIEFS**

SIGNS OF THE TIMES



by Laura Chen and Barbara Plocinik

YOU see them along highways, at airports and bus terminals, and where you work. They direct you to the telephone, the restroom, or a night's lodging. They can warn you of danger or death, but they can also mislead you!

What are these things that can be both helpful and confusing? Signs.

A sign exists for one purpose—to communicate its message clearly to everyone who sees it. However, confusion often arises because there is no language that all people understand. To circumvent this barrier, more and more groups are substituting pictorial symbols for written signs.

Although Europeans have used pictograms, as these symbols are known, since the early 1900's to convey traffic information, it is only in recent years that they have been put in buildings to guide, protect, or inform occupants and visitors. And now, in the United States, symbols are used increasingly for transportation systems, hazard warnings, worker safety, and fire safety, as well as for public information.

Pictograms are extremely valuable in communicating important information to the 8 million Americans for whom English is not the native language and to the estimated 2 to 40 million adults who are functionally illiterate. Another advantage of pictograms is that, in some cases, people understand them more rapidly and accurately—and from greater distances—than word signs.

This growth in the use of pictograms has, however, led to a new problem—the widespread proliferation of confusing and even contradictory symbols. Each person, agency, or industry that feels the need for a symbol develops one, often without consulting any other group or evaluating the symbol's effectiveness in communicating the intended message.

To provide a basis for eventually standardizing symbols, scientists in the Building Safety and Security Group (BSSG) of the NBS Center for Building Technology are developing evaluation methods to determine what makes one symbol more effective than another. In cooperation with the National Fire Protection Association (NFPA), group leader Brian Pierman and research psychologists Drs. Belinda Collins and Neil Lerner have been testing fire safety symbols proposed by the International Organization for Standardization (ISO).

Using placards, they presented the proposed symbols to volunteers from a community college, a retirement home, and several fire stations. The subjects were asked what each symbol signified to them.

The BSSG researchers then used other testing methods to check the validity of their results. In addition to the placards, the symbols were presented on slides and in booklets to other groups of volunteers who were asked to write down definitions while others took a multiple choice test. The subjects were also asked to indicate, on a scale from 1 to 5, their confidence that they had selected the correct answer. The results showed no significant differences between the various presentation methods. While subjects performed somewhat better with the multiple-choice answers, the ordering of the results was similar for both multiple choice and definition answers.

The survey did reveal, however, that there would be serious drawbacks if some of the proposed symbols were used immediately in the United States. For example, only 25 percent of the subjects correctly identified the proposed ISO exit symbol while just 20 percent understood the "do not block—keep passageway clear" symbol.

According to Pierman, the most serious problem with a symbol arises when a person interprets its message to be the exact opposite of what is intended. Since the penalty for misinterpretation of such a sign in an emergency situation could be serious injury or death, the need for proper evaluation of such symbols is obvious. In one NBS study, for example, 140 of the 143 subjects either misidentified or did not respond to the proposed "no exit" symbol. Even worse, the majority who incorrectly identified the symbol interpreted it to mean "exit" or "safe area." NBS recommendations to ISO have prompted revisions of the proposed "exit" and "no exit" symbols.

In addition to their continuing research on fire emergency symbols, the NBS group is testing and evaluating safety symbols in the workplace—another area where effective communication is vital. According to the Labor Department's statistics, in 1978 more than 5.6 million people were injured and, in workplaces with more than 11 employees, about 4500 people were killed in work-related accidents in the United States. The most frequent causes of these accidents were ones that could have been avoided or eliminated, such as work areas not kept clean or orderly, improperly maintained equipment, clogged aisles, unmarked or blocked exits, unposted "no smoking" areas, and the failure of employees to

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Chen is a DIMENSIONS staff writer and Plocinik is a former editor of DIMENSIONS/NBS.

Researchers Brian Pierman, Belinda Collins, and Neil Lerner of the NBS Center for Building Technology discuss two of the many safety symbols they are studying to determine what makes one symbol more effective than another.

wear hard hats, safety goggles, or other protective equipment where dangers existed. Effective signs that remind or alert employees to these hazards could help lower these statistics.

Consequently, the National Institute of Occupational Safety and Health (NIOSH) has asked NBS to conduct research on effective, consistent symbols for workplace hazards. The findings will also be made available to the American National Standards Institute (ANSI), a voluntary standards-setting organization.

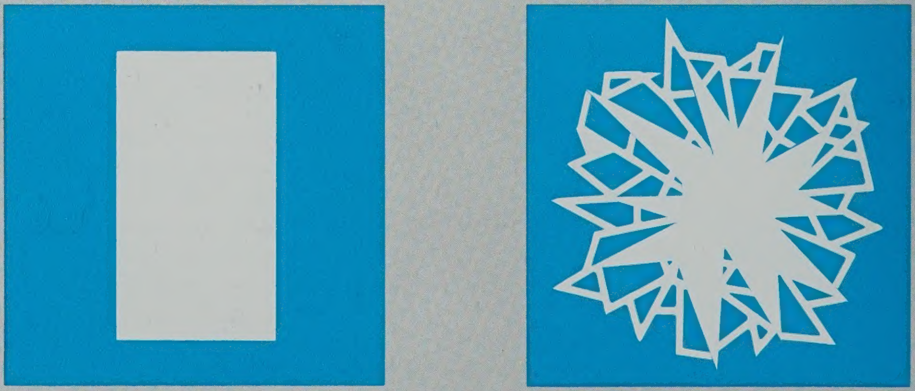
According to Collins, chairperson of ANSI's subcommittee on safety symbols, the evaluation of workplace safety symbols will require three main steps. "First we must determine which types of situations require symbols. What messages need to be communicated and for which hazards? Secondly, we will select a set of candidate symbols for each message. Finally, we will evaluate the proposed symbols to determine which ones best communicate the desired meanings and make our recommendations to NIOSH."

In the first phase of this project, Collins and Lerner visited industrial plants of various types and sizes including a truck engine factory, a ceramic manufacturer, an aircraft assembly plant, and a chemical company. They found that the plants were using many different signs and symbols to convey the same message—a practice that can only lead to confusion, not safety. They also talked with workers, plant safety officers, and managers to assess the effectiveness of the signs. In addition, they reviewed existing symbols from sign catalogues, manufacturers, and national and international standards.

The researchers are now in the process of selecting three images for each safety message under study. They will then evaluate the new symbols for their effectiveness in conveying the intended message. The group also plans to assess parameters such as smoke, stress, and distance, that can affect symbol detection, along with other critical factors including attention-getting ability, placement, and behavioral effectiveness.

Another group of NBS scientists is investigating various conditions that affect people's perception of color in safety signs. A new set of problems in chromatic adaptation has resulted from today's increased emphasis on the use of energy-efficient light sources. For example, the inefficient incandescent light bulb that has been in use for the past

These are some of the symbols NBS researchers have asked volunteers to rate. What do you think they are intended to communicate? See page 6 for the answers.



100 years emits energy throughout the visible region of the spectrum (400 to 700 nm). It produces energy in wavelengths which we associate with the reds, oranges, yellows, greens, and blues with which we are so familiar. On the other hand, the low-pressure sodium lamp which is 9 times more efficient than its ancestor emits most of its energy in an extremely narrow range—589 to 589.6 nm—producing a yellow-orange light and causing colors viewed under it to appear quite different from the way they would under incandescent light.

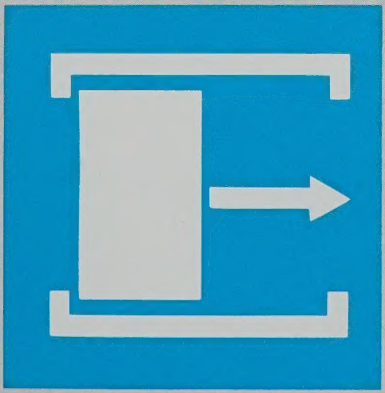
Sponsored by OSHA, this research has significant safety implications. As Dr. Robert Glass, chairperson of the Research Subcommittee of ANSI's Visual Alerting Systems Committee states, "Red in a sign means that you can lose your life if you don't heed the sign. . . . It is therefore important to study the visual sensitivity of a person's eyes in different environmental conditions, or before and after a person has adapted to a different light to determine how that person perceives colors under different conditions."

To study color distortion and its related safety problems, Glass and Dr. Gerald Howett, experimental psychologists in the NBS Sensory Environment Group, and Marvel Freund, an engineering technician in the Radiometric Physics Division, developed two special laboratories—the Color Applications Laboratory and the Illumination Color Laboratory.

The Color Applications Laboratory, a "basic research" facility, consists of a multi-channel electro-optical system that allows light to be delivered to the human eye in an experimentally reproducible manner. Glass designed the NBS optical system to allow exact control over the wavelength, intensity, size, pattern, spectral energy distribution, and flash rate of the stimulus that a person's eyes will receive. Using this system, the amount of chromatic adaptation in humans can be measured.

To test the effectiveness of different symbols, the NBS researchers present symbols on either placards, slides, or in booklets to volunteers to see how they interpret the symbols.

The other facility, the Illumination Color Laboratory, was created to provide a realistic environment for studying color distortion when an object or area is illuminated by one of the more energy efficient types of lighting now available or being developed. This "applied" laboratory is constructed with changeable walls in order to vary colors and reflectance values. Six wall colors are currently available for use: red, blue, green, gray, white, and black. Two layers of translucent plastic diffusers form the ceiling of the chamber and allow for evenly distributed illumination.



Experimental psychologist Robert Glass is shown below making adjustments on the multi-channel electro-optical system he developed that allows light to be delivered to the human eye in an experimentally reproducible manner. The Illumination Color Laboratory at the right allows Glass to observe the appearance of different pigment compositions under various energy-efficient lights.

The lab allows the researchers to study the appearance of different pigment compositions on colors. For example, the familiar red color appears gray under a low-pressure sodium light, whereas a fluorescent red appears as we expect. This occurs because the fluorescent red absorbs light at 589.6 nm (yellow-orange in the visible electromagnetic spectrum) but emits the energy at a lower wavelength causing it to appear red.

The scientists have studied the appearance of red paint under various lights. They found that energy-efficient low-pressure sodium lamps make red appear gray, high-pressure sodium lamps make the red paint appear more like orange while an uncoated mercury light source makes the red paint appear more like brown. The NBS group is examining the feasibility of mixing energy-efficient lights that distort colors with less efficient sources such as fluorescent and tungsten lights that are high in color rendering. Such a mixture could maximize color rendering while minimizing energy consumption.

The meanings of the symbols from pages 4 and 5 are:

1. emergency exit
2. break glass to obtain access
3. blind alley
4. slide door to right to open

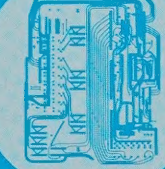
Using the two labs, the scientists can simulate a real working condition in the Illumination Color Lab and then test for eye responses in the Color Applications Lab. A typical experiment might have a subject carry out office work under low-pressure sodium lights all morning. Then the researchers would test the worker's eyes to see when the subject would see colors normally. Chromatic adaptation would be especially important if a worker spent a long time under an energy-efficient light in one part of a building and then went outside in a hazardous area where he or she would need to see colored warning signs.

Based on their findings, the NBS group will make recommendations to OSHA to ensure that the appropriate safety colors are seen under different light sources. OSHA is responsible for setting mandatory standards for particular colors used on warning and danger signs in the U.S. workplace.

The Occupational Safety and Health Act of 1970 requires every employer to provide a job environment that is free from recognized hazards that can cause death or serious injury. NBS research to develop and standardize safety symbols and to determine the conditions necessary for people to see these signs properly will not only help employers meet their legal obligation but, more importantly, it should help keep employees from being needlessly injured on the job. □

This text has been edited from the keynote address given by Dr. Thomas A. Dillon, Deputy Director of the National Bureau of Standards, at the Measurement Science Conference in San Luis Obispo, Calif., November 30, 1979.

Measurement & Development in the 1980's

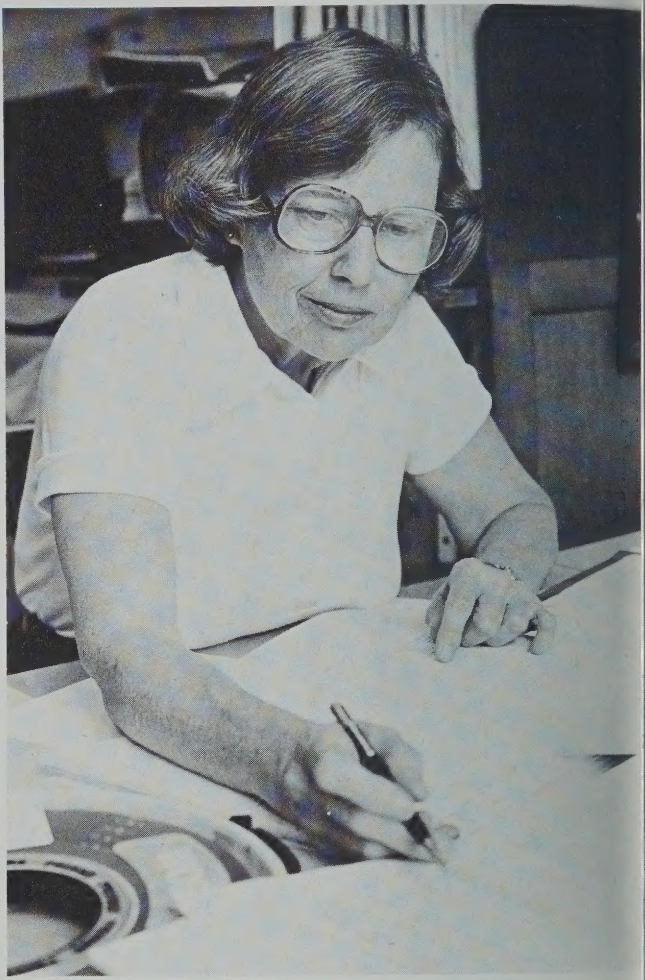


by Thomas A. Dillon

THE economic and technological environment in this country seems ready for a major surge of industrial development in the eighties. Recent scientific breakthroughs including lasers, semiconductors, computers, and new materials are ripe for full industrial exploitation. However, the measurement science needed to fully support such a surge has not been completely developed. Many technological and institutional challenges remain before us, and business, industry, academia, and government all have important roles to play in what President Carter says "must be a continuing battle to maintain the technological strength of the American economy."

Innovation and economic growth may be difficult in light of inflation problems, but I'm convinced that the measurement community can make important contributions. Measurements, process control,

Thomas A. Dillon, Deputy Director of NBS.



Mathematician Eloise Evans is one of several research associates sponsored by the International Centre for Diffraction Data (ICDD) working at NBS. The group, in cooperation with Bureau researchers, produces highly accurate patterns for the Powder Diffraction File, which is maintained by ICDD and used by businesses, universities, research laboratories, and law enforcement groups from around the world.

quality assurance, and reliability are areas where productivity leverage is possible, often at low costs. Electronics is one area that presents many such opportunities.

Every facet of modern life, from defense to recreation, is affected by electronics. Business Week said of semiconductors that "perhaps no other industry is so important to the future economic health of the Nation."

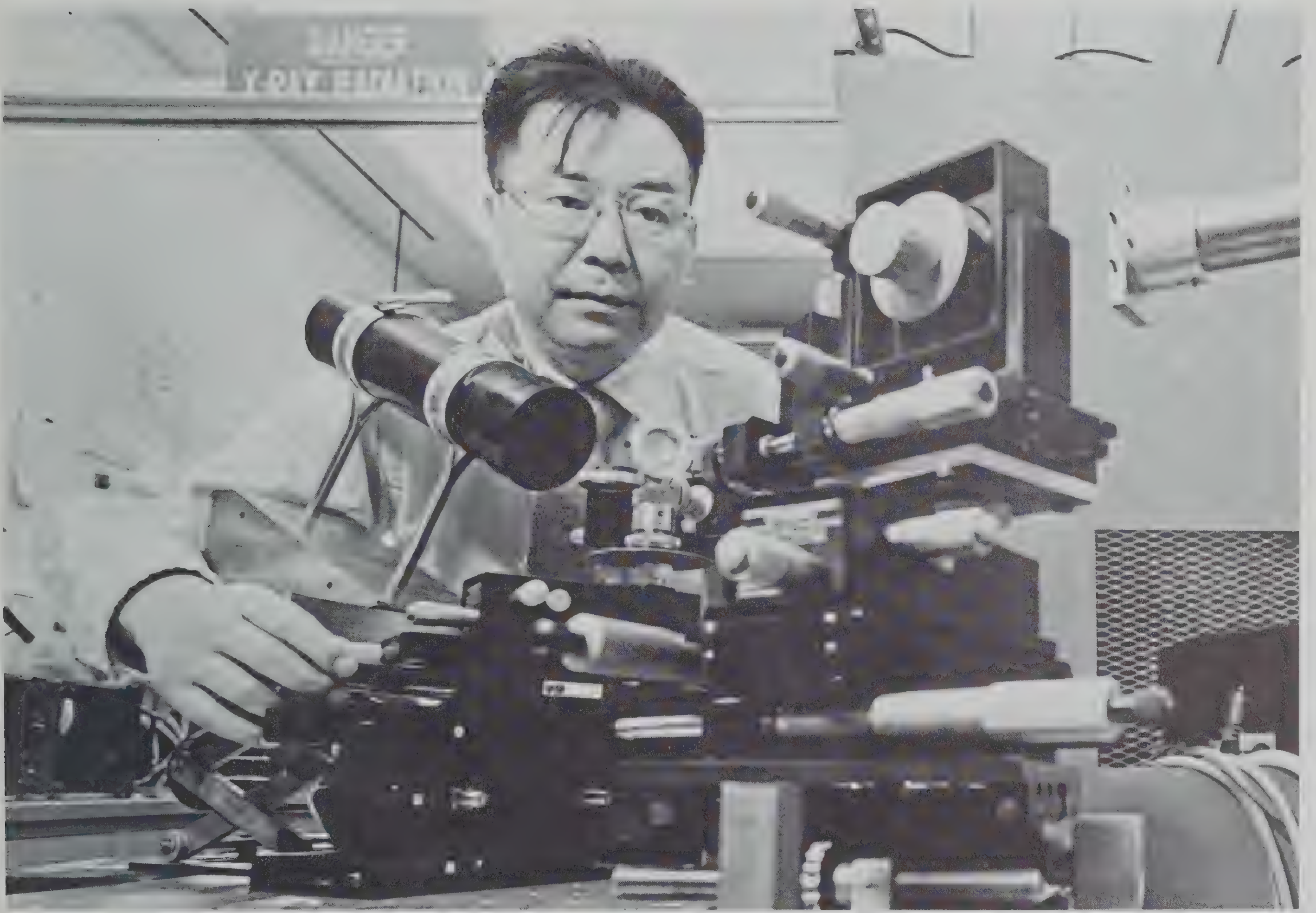
Unfortunately, our worldwide dominance of the semiconductor field is eroding. Both the Semiconductor Industry Association and the Electronic Industries Association have expressed their alarm to Congress. They worry about the threats to U.S. leadership from numerous foreign government-industry consortia. If further losses are to be avoided, we must produce reliable, very large scale integration (VLSI) devices at low costs. Doing so requires a better understanding of the materials involved and better control of the production process.

NBS is ready to help in this area, and we are planning a major program in VLSI. This will be an extension of our long-term, highly successful program in electronic reliability. Program elements include wafer characterization, interface evaluation, on-line quality control, test structures for VLSI geometry, testing protocols, and package evaluation.

NBS also has a major effort underway to help solve some of the problems in automated manufacturing. Our intent is to produce standards, data bases, and new measurement and control technology. Towards this end, we have recently adapted a solid-state camera to a 6-axis robot arm. Information about the position of the workpiece is sent from the camera to a computer, which in turn guides the arm to the object. This vision capability greatly expands the usefulness of the robot and may permit elimination of costly transfer lines that ensure accurate placement of the workpiece.

While working on these projects, NBS researchers maintain close contact with the Robot Institute of America, the Numerical Control Society, and the Society of Manufacturing Engineers. We also coordinate very closely with the Air Force's integrated Computer Aided Manufacturing Program. Our goal is to ensure that automation technology is made accessible to the small batch manufacturing sector. The productivity gains are potentially enormous.

Materials are another large factor in our economy—accounting for about half the gross national product. As the costs of traditional materials increase and as additional demands are placed on materials, new, innovative materials may be substituted. In many cases this will increase productivity, improve performance, reduce weight, and



An NBS research team led by physicist Masao Kuriyama has developed a device and technique for nondestructive evaluation that magnifies x-ray images on a real-time basis with significantly improved image resolution.

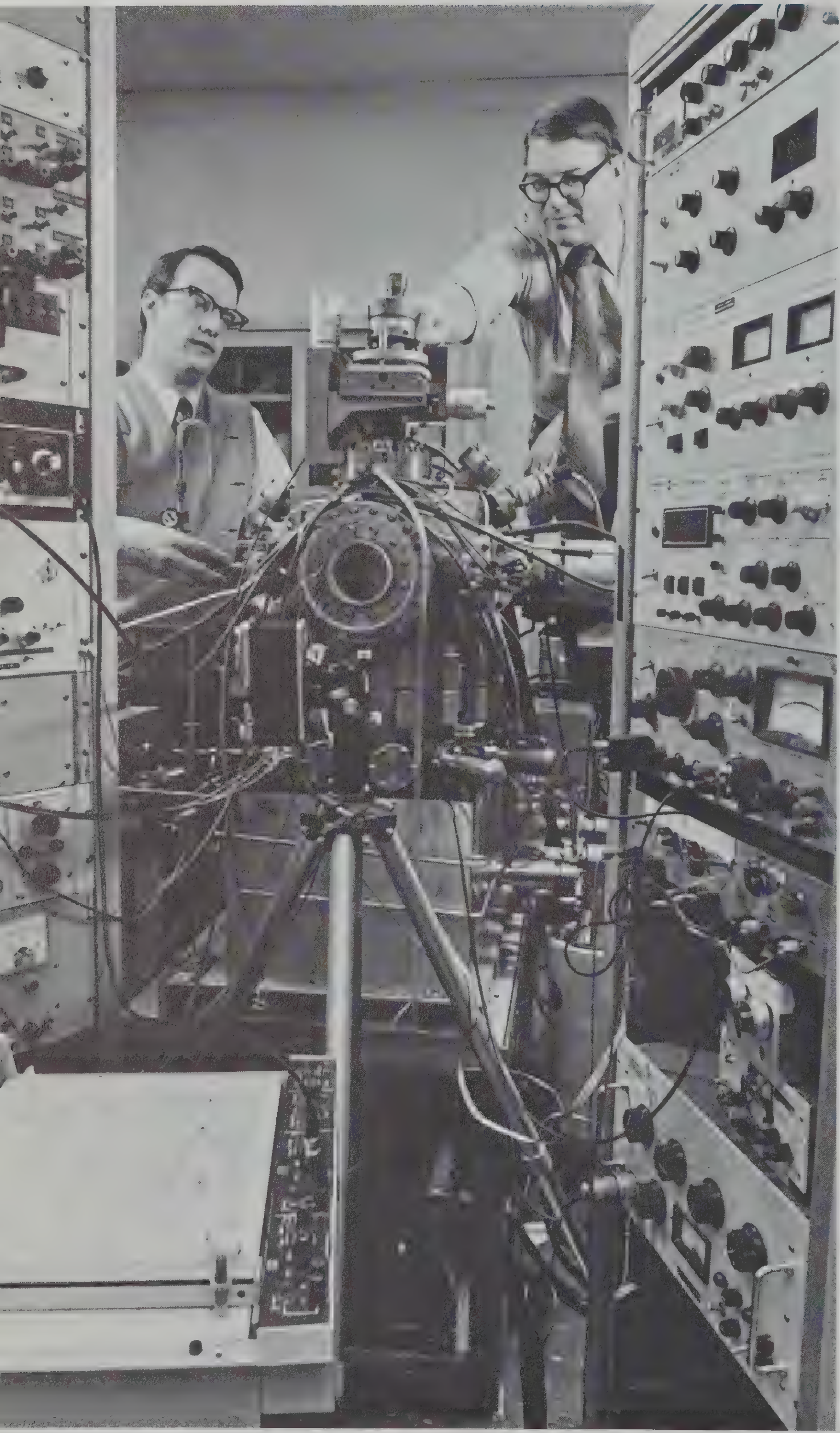
at the same time, decrease our dependence on foreign sources of supply.

But the use of new, high-performance materials presents problems in the areas of measurement and quality control. Modern technology requirements push our most sophisticated materials to their limits of performance. Under such circumstances, even small imperfections in microstructures cause an unacceptable loss of performance. Turbine blades for jet engines, for example, are used at such high temperatures that inclusions and small changes in microstructure cannot be tolerated. This puts a great premium on industrial quality control. Examination of materials with nondestructive techniques capable of detecting minute flaws is vital during the manufacturing process.

Responding to these challenges, NBS is developing a more thorough fundamental understanding of alloy solidification, a prerequisite to the control capability needed for powder metallurgy, glassy metals, dual phase steels, and advanced casting processes. Of course, we plan to continue our current programs in nondestructive testing using x-rays, neutron radiography, acoustic emission, and ultrasonic imaging.

Another problem affecting the quest for higher productivity is that of rapid measurement. More and more industries are turning to automated test equipment, and annual sales of such equipment are expected to top $3 billion by 1981. This investment is repaid by higher productivity only if the measurements are reliable.

This area may prove to be the ultimate challenge for measurement science. The speed requirements and intrinsic complexity may necessitate "built-in or self" assurance, coupled with on-site tests of the absolute overall system performance. Truly a formidable measurement task.



Electron Stimulated Desorption Ion Angular Distributions (ESDIAD) is a method for directly determining surface molecular structure. Reseachers Theodore E. Madey (left) and John T. Yates, Jr., are shown with the ESDIAD apparatus, one of several measurement tools developed in the Bureau's surface science program.

We at NBS have several projects underway which address some of these concerns. They include:

—A calibration system for analog-to-digital-convertors,

—A test method for sample/hold amplifiers,

—Development of picosecond pulse measurements, and

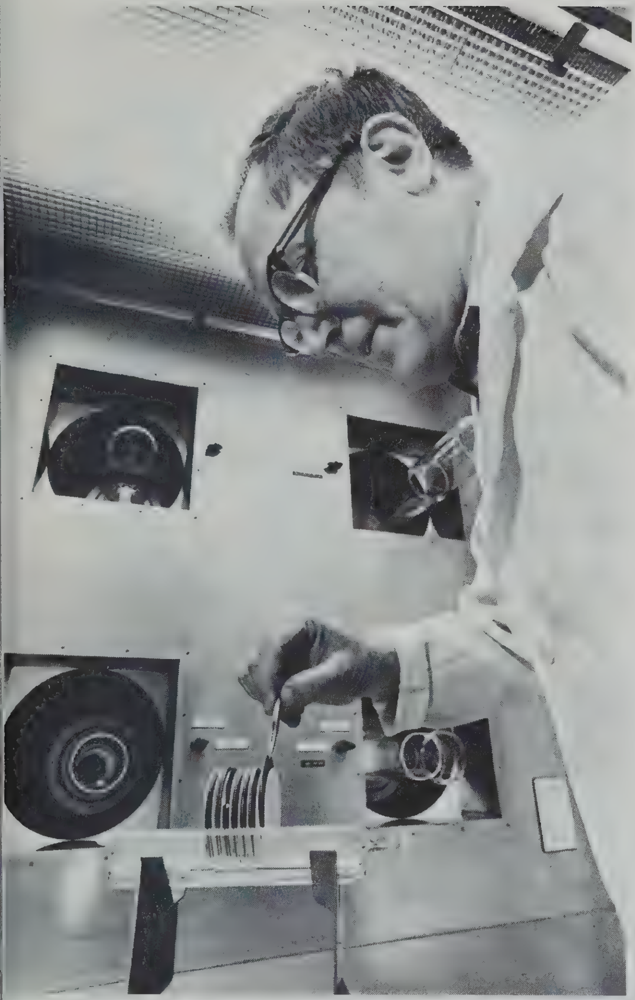
—Research on Josephson-junction devices, including a 4-bit analog-to-digital convertor that operates at 200 megasamples per second.

So far I have focused on some of the technological and institutional challenges the measurement community faces as we enter into the eighties. In terms of industrial development and economic strength the stakes are very high. Now I'd like to shift gears and discuss some ideas for meeting those challenges. Let's look at the scientific and technological opportunities that promise real advances in measurement science.

The first of these is the microprocessor. The availability of inexpensive programmable microprocessors is already revolutionizing measurement science, and this revolution will accelerate in the 1980's. The automatic test equipment I mentioned earlier is, of course, dependent on the microprocessor. So is automated batch manufacturing. Microprocessors make it possible to accumulate and analyze large volumes of data, and to use the measurement results in feedback and automatic control.

Another area in which NBS is very active, and which promises major pay-offs for the 1980's, is surface science. Understanding surfaces is important in catalysis, corrosion, lubrication, and wear and in technologies such as microelectronics. To understand surfaces requires accurate measurement of many quantities, ranging from bond geometry to charge distribution, or from thin films to reaction intermediates. For many of these quantities no method exists for accurate measurement. The NBS program includes basic theoretical studies, a variety of measurement techniques for probing the surface with electrons, ions, and neutrons, and techniques to monitor absorbed and desorbing species using infrared and visible spectroscopy.

We are developing strength in organic electrochemistry. New theories, insights, equipment, and



Part of the Bureau's program in electronic reliability is aimed at developing methods for testing tiny electronic circuits. Here NBS technician John Krawczyk places specially treated silicon wafers in a high temperature diffusion furnace, one step in the production of a semiconductor test pattern.

approaches have brought electrochemistry to a stage of rapid development. NBS will use this capability to develop methods for identifying and measuring organic materials, as well as for studying basic reaction mechanisms and kinetics. Specific program plans call for spectrochemical studies of oxocarbons, a computer-controlled fast potentiostat, and novel approaches to electrochemical detection. This knowledge will have a substantial impact on the understanding of biochemical, environmental, and energy conversion processes.

The list goes on and on. Modern computers teamed with new mathematical techniques may allow us to model and predict complex, non-linear phenomena such as turbulence. Picosecond pulse lasers may permit the probing of molecular systems on a time scale comparable to the chemical reaction itself.

However, the magnitude of the measurement community's contribution to economic development will depend on cooperation—cooperation among universities, industry, Federal agencies, and State and local governments.

At NBS we have established specific mechanisms to accomplish this. For example, our industrial Research Associate Program brings scientists from industry to work at NBS for a year or two. The research associates, paid by industry, cooperate with Bureau scientists on projects of mutual concern. There are currently 90 research associates at NBS, and we welcome suggestions for new programs of this type.

We also have ties with academia. Through the Post-Doctoral Research Program, top graduates join us for a year or two of research experience. In addition, many university professors collaborate with NBS scientists on research projects. These projects often involve unique NBS facilities such as the reactor and cyclotron.

Through the Intergovernmental Personnel Act, people from any level of government can come to NBS and work on mutual problems.

We work very closely with such groups as the National Conference of Standards Laboratories and the National Conference on Weights and Measures. And we are deeply involved in the standards-writing committees of the American Society for Testing and Materials, the American National Standards Institute, and dozens of other organizations.

These NBS partnerships help us sharpen the definition of our programs, bring new ideas and perspectives to bear on solving measurement problems, and expedite dissemination of research results.

Let me close by summarizing very briefly. Measurement science can make crucial contributions during the eighties, pervading almost every vital concern of our complex society. Industrial development and the economy are particularly good examples of where measurement could have substantial impact. However, there are real challenges facing the measurement community which may limit the benefits. At NBS we look forward to the coming decade and to cooperating in a broad, working partnership that will take advantage of scientific and technological opportunities to meet those challenges. □

COVER
STORY

"Thousands of barnacles small and great
Stick to the jolly old ship of State,
So we mustn't be cross if she seems to crawl,
It's rather a marvel she goes at all."
A. P. Herbert

THE END OF THE FREE RIDE

MAKING ANTIFOULING COATINGS THAT LAST LONGER

by Gail Porter

FROM the day the first ship's hull slid into seawater, small crustaceans with the demeanor of mules have followed. Commonly known as barnacles, they are among the most tenacious of a group of marine life known to sailors as fouling organisms. These animals find a ship's hull an accommodating home and are consequently a major

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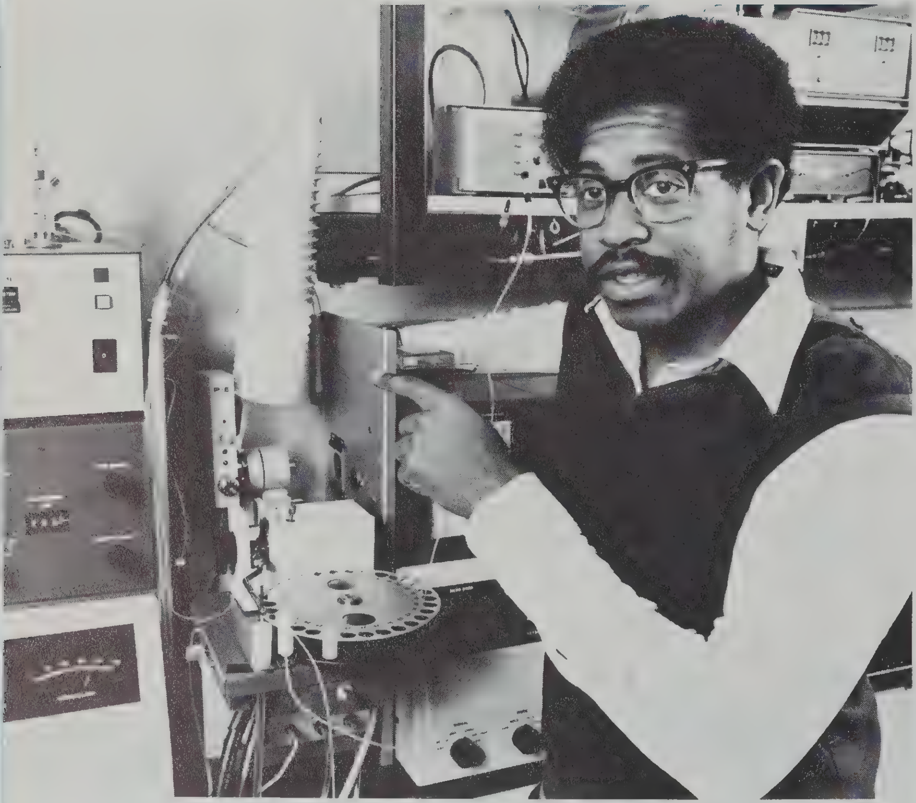
Porter is a writer and public information specialist in the NBS Public Information Division.

cause of hull drag which reduces the speed and fuel efficiency of ships.

Over the centuries, crafty seafarers have tried their best to outwit these unwelcome freeloaders. As early as the 12th century B.C. Phoenician seatraders covered the hulls of their boats with copper to reduce fouling. The Greek inventor and mathematician Archimedes is reported to have sheathed the bottoms of his ships with lead. The fleet of Henry VIII carried a "protective" covering of animal hair attached with pitch, while throughout the 1800's and 1900's patents were issued for all sorts of antifouling concoctions, including one described as "grease from boiled bones, kitchen stuff and butter without salt, mixed with poisonous materials."

None of these methods was entirely effective as evidenced by the fact that before World War II it was not uncommon for a single ship to be drydocked with as much as 300 tons of these organisms clinging to its hull. The drag resulting from such fouling can increase the fuel consumption of a ship by as much as 50 percent.

During World War II, improved hull paints containing cuprous (copper) oxide as a biological toxicant were developed by the Navy as a strategic weapon to lengthen the time ships could be at sea between drydockings. Derivatives of these copper-based paints as well as less widely used organotin-based paints are still the most common protective coatings applied to Navy ships. These paints, generally effective for 24 months, are considered to be the best antifoulant materials available. But, even with these coatings, by the time a ship is drydocked after an average 5 years at sea, the Navy estimates that it uses 20 to 30 percent more fuel because of drag from fouling. While average costs for hull scraping and repainting are difficult to isolate from other drydocking costs, the increased fuel costs for



Inorganic chemist Kenneth Jewett describes the operation of atomic absorption instruments he uses to identify specific compounds which leach from the coatings.

the Navy's fleet amount to an average of $50 million per year.

However, a new type of experimental coating may soon provide Navy ships with much better fouling protection. These coatings, developed at the Naval Ship Research and Development Center at Annapolis, Maryland, consist of a plastic to which biocidal organometallic compounds have been chemically bonded. According to test results so far, the most effective of these metal compounds appears to be tri-n-butyltin and/or tri-n-propyltin. The bonding of the tin to the polymer backbone causes these coatings to last much longer than copper-based antifoulants.

The copper- and tin-based paints now in use release the toxic material to the surrounding water rather quickly because the biocide is simply mixed with a paint base. In contrast, as the experimental organometallic polymer (OMP) coating sits in water, the bonds between the tin and its plastic substrate are broken gradually, releasing the biocide in a much slower fashion—thus providing protection for a longer period of time with less risk of environmental pollution.

The OMP coatings are designed to protect a ship for the full 5 years between drydockings. The first real test of their effectiveness began last year when two Navy ships, one based in Pearl Harbor, Hawaii, and the other in Charleston, S.C., were coated with test strips of the polymer for a 5-year trial.

In the meantime, the Navy has asked the National Bureau of Standards to help them learn more about the behavior and molecular structure of these special materials. "The Navy came to us," says Frederick E. Brinckman, a group leader in the NBS Chemical Stability and Corrosion Division, "because we were already familiar with this area."

For over a decade, Brinckman and NBS biologist Warren Iverson have been studying the characterization and measurement of materials degradation in water. These types of processes are very important in the study of the corrosion of metals, as well as for intended chemical breakdowns such as the release of biocides from antifouling OMP coatings.

The deterioration of metal-containing materials in natural environments such as seawater, says Brinckman, is not always a simple chemical reaction. In many cases microscopic life forms actively break down materials and in some cases even transform their components into toxic chemical forms which can enter food chains.

As recently as 10 years ago, he says, scientists believed that heavy metals like mercury, lead, tin, and cadmium could not be metabolized by microorganisms. It was thought that when industrial pollution containing these metals entered the water system, the metals simply precipitated and were subsequently buried in the sediment. The discovery of abnormally high levels of methylmercury in fish proved this theory to be wrong. Since then biologists have identified certain strains of bacteria that can transform metal compounds into methylated forms, which are easily absorbed by animal tissue and are extremely toxic at very low levels of exposure.

Realizing the importance of this area of research, Brinckman and Iverson began forming an interdisciplinary research team which was later christened the Chemical and Biodegradation Process Group. The Group's goal was to measure very carefully the interaction between the chemistry of metals and the biology of microorganisms. "We were no longer just looking at how our materials do in the real

Physical chemist Jo-Anne Jackson examines chemical spectra displayed on an oscilloscope screen. While a mass spectrometer differentiates molecule fragments in a sample, a computer stores and interprets the data for future use.

world," says Brinckman, "but also at how the world is affected by our materials."

A key element in the Group's program was the development of methods to differentiate between various kinds or species of trace metal compounds, a task encompassing elements of the diverse disciplines of electromicrobiology, spectroscopy, and organometal chemistry. These techniques were then applied to a wide spectrum of environmental and materials durability problems from the accumulation of metal pollution in the harbors of the Rhine River estuary in Holland to the soil degradation of methylarsenic-containing pesticides used on the U.S. cotton crop.

So when the Navy asked for help characterizing the molecular structure and defining precisely the leaching properties of their antifouling polymer, the Bureau was aptly suited for the job.

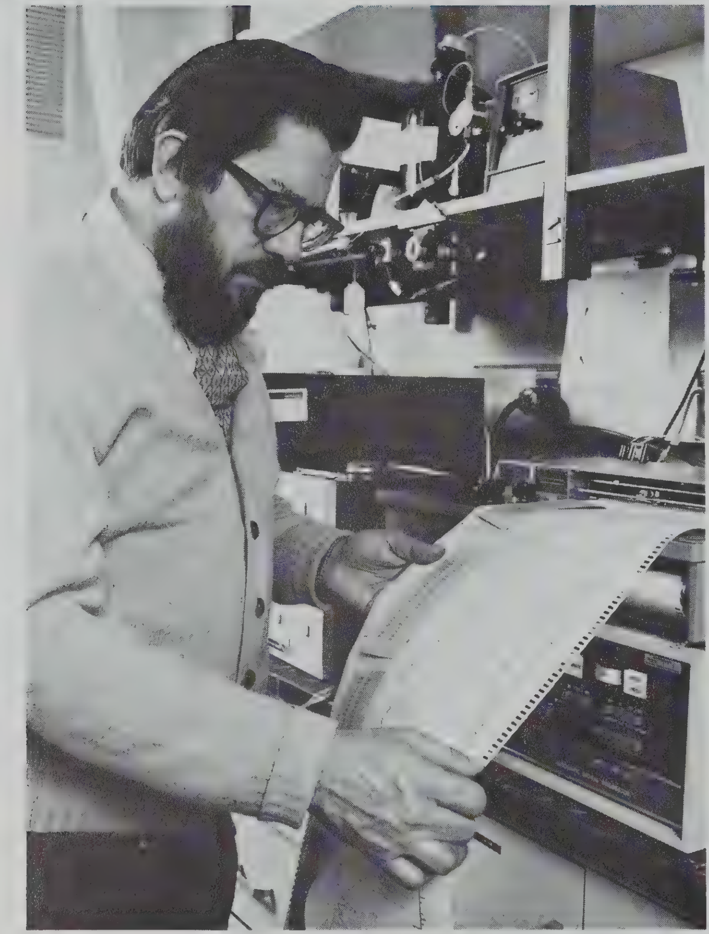
"We are measuring the leaching rates," explains Iverson, "to help in developing more effective formulations. If it (the tin compound in the polymer) leaches out too fast, it's not going to last very long. On the other hand, if it leaches out too slowly, it won't be effective."

"In seawater," points out Brinckman, who heads this project, "there are 1000 to 10 000 living cells per milliliter." Many of these are slime-forming bacteria which very rapidly attach to surfaces. Once a thin film of bacteria has taken hold, fungi and algae begin accumulating, followed by grasses and finally, barnacles, oysters, and other higher level shelled animals.

The idea behind the antifouling coatings, he says, is to "target the precursor organisms to prevent the whole chain of events." The problem is to develop a coating which selectively inhibits the slime-forming bacteria without endangering other forms of sealife or causing the toxic tin compounds to accumulate in the food chain, creating a health hazard.

This is why knowing the rate of release of the tin compound into the surrounding water is critical for evaluating the effectiveness and safety of the coatings. Also, while the Navy knows which types of experimental coatings seem to be the most effective in preventing marine fouling, Navy researchers would like to know *why* some polymer types are superior to others. Why for example do some polymer formulations allow the tin to leach at a much slower rate than others?

Microbiologist Warren Iverson draws a sample of air from a test tube containing Pseudomonas bacteria to determine if the bacteria can metabolize and respire biocidal tin compounds.



Organic chemist Edwin Parks reviews the data from his gel permeation chromatography analysis of the Navy's organometallic polymer coatings.

Part of the reason, according to organic chemist Edwin J. Parks, may be that some polymer samples contain a higher percentage of the tin compound in the higher molecular weight fraction of the polymer. In other words, proportionately more of the biocide is bonded to the larger, heavier, polymer molecules. Generally, says Parks, high molecular weight fractions of the polymer release their tin components more slowly and at more predictable rates than do lower weight fractions.

During the past year, Parks has examined a number of coating samples provided by the Navy. The methods he uses to characterize their molecular structure and to locate the tin compounds combine a variety of different analytical techniques.

The first of these, gel permeation chromatography, is a sophisticated procedure based on a rather simple fact—that is, high molecular weight components in a polymer mixture are larger than lower weight components. Samples of the polymer coatings are passed through a stainless steel tube or column containing a porous packing material. The packing in the column separates the polymer into molecular groups of different sizes. The packing substance is "permeated" with indentations large enough to "catch" the smaller molecular groups but too small to catch the larger types. The result is a separation of the fractions, because the larger molecular groups move through the column more quickly.

Analyzing the separated fractions, Parks is able to quantify the amount of organotin bonded to each fraction, using detection methods such as ultraviolet or atomic absorption spectroscopy. Ultraviolet absorption spectroscopy is based on the ability of certain molecular groups to absorb ultraviolet light of specific frequencies. Changes in the ultraviolet absorption patterns of the different fractions as they pass through the column indicate the nature and amount of these molecular groups.

Small samples of the liquid drawn with an automatic pipette are then analyzed with atomic absorption spectroscopy to determine the amount of tin bonded to each group. With a burst of heat from a minifurnace, reaching temperatures of almost 3000 °C, all chemical bonds in the liquid are effectively broken. Light at a specific and characteristic wavelength from an ionized gas lamp containing tin atoms is passed through the resulting hot furnace atmosphere.

Any tin atoms present in the atmosphere will absorb some of the light which is specific to that element. The amount of light absorbed is an indication of how much tin is present in each sample.

In addition to determining where the tin is bonded to the polymer, the researchers need to know exactly what type of tin-containing compound leaches from the polymer coatings. According to inorganic chemist Ken Jewett, the Navy has already estimated the total amount of tin released by specific polymer samples under conditions simulating use. What is needed is a more sensitive analysis of the leaching rates and more information about the way tin compounds break away from the polymer. Does the whole compound or just one segment of it break off? This information is important for predicting the environmental impact of the leached toxicant, as well as for determining which type or combination of tin compounds will make the most effective biocide.

At this stage of his work with the Group, Jewett is establishing baseline leaching data for the Navy by using ion exchange chromatography and atomic absorption spectroscopy. The combination of these two very sensitive techniques allows NBS researchers to detect as little as 10-50 nanograms of a particular kind of organotin molecule in seawater—that's equal to about 50 *billionths* of the weight of a dollar bill.

But the question remains, what happens to the tin after it leaches from the polymer base? Are some tin compounds more likely to be harmful to the environment than others? Are there certain types of bacteria that can tolerate the biocide or that can transform the tin compounds into more toxic methylated forms?

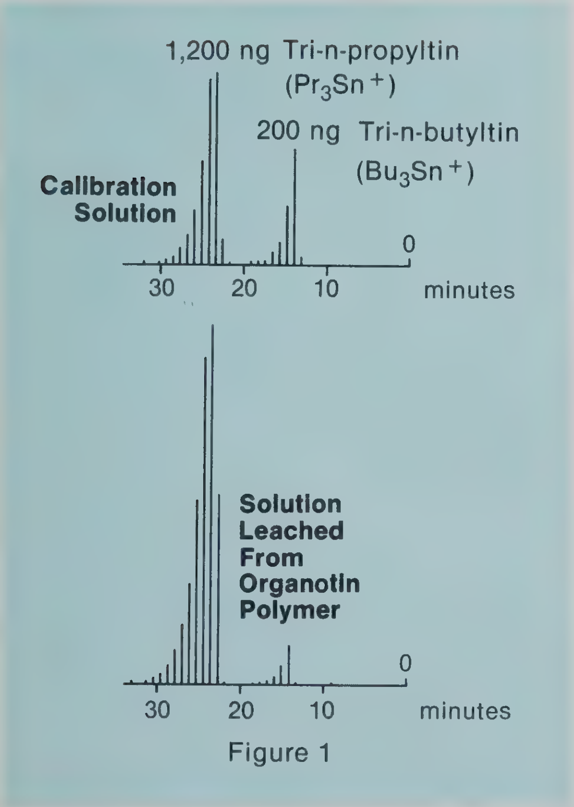


Figure 1—*Graphs from an atomic absorption analysis of liquid chromatograph elements show that the biocidal ionic species tri-n-propyltin and tri-n-butyltin leach from the organometallic polymer coatings without breaking apart. The upper graph shows the absorption peaks and concentrations of a known solution containing the compounds. This is matched with the lower graph to confirm the identity and to establish the concentration of the compounds in the leached solution.*

Iverson and chemical biologist William Blair are testing certain strains of *Pseudomonas* bacteria, previously identified by NBS research as resistant to metals, to see if the bacteria can transform the tin compounds used in Navy polymer coatings. Samples of the bacteria are allowed to grow on agar surfaces treated with the various tin biocides under study. The atmosphere above the bacteria is then analyzed with several combinations of gas chromatography, flame emission spectroscopy and mass spectrometric techniques to see if the bacteria are metabolizing the tin and exhaling it in some other form.

The researchers will soon be doing similar tests with mixed cultures of common marine bacteria in an effort to measure directly the efficiency of the biocide on other types of slime-forming bacteria. Combining these two sets of data will tell them which compounds pose the least danger to the environment while providing the best antifouling protection.

The common factor needed in all of these experiments—polymer characterization, leaching properties studies, and biological tests—is extremely accurate measurement and identification of specific chemical compounds, often at trace levels equivalent to environmental concentrations (parts per billion).

As the Nation's primary measurement laboratory, NBS has taken a leading role in the development of the delicate techniques needed for these kinds of studies. The instruments used in chromatography, spectroscopy, and mass spectrometry are rather like violins which must be sensitively played depending on the task at hand. "It may take months" explains Iverson, "to come up with the best methods for detecting a particular compound."

With the help of these exacting measurements, the Navy may be able to refine its polymer coatings into the first truly effective, long-lasting antifoulant. "The basic technology developed may also be applicable," says Brinckman, "to other time-release mechanisms important in medicine, fossil fuel recovery, or materials protection problems such as wood preservation." Moreover, the accurate characterization of these coatings will allow the Navy to write better specifications for manufacturers of antifouling coatings who are interested in supplying the materials once the Navy is ready to have them produced in bulk.

It all spells a rather dim picture for future generations of home-hunting barnacles. But for Navy ship captains it may mean the victorious end to a sea battle centuries old. □

SPECIAL

Improved X-ray and γ-ray Measurements Have Several Applications

by Richard D. Deslattes

THE NBS mandate to maintain and improve the basis for physical measurement is realized in many ways. Among several groups at NBS working toward unification of the entire electromagnetic spectrum, ours, the Quantum Metrology Group, has been particularly active in the areas of x-ray and γ-ray measurement. Our efforts had, in past work, improved the direct measures of the Avogadro constant (i.e., the conversion between the atomic mass scale and the macroscopic, kilogram scale*).

In a continuing experimental effort, our group has now significantly improved the accuracies with which certain γ-ray transitions and x-ray transitions are coupled to optical wavelength standards. The optical reference source was chosen to be compatible with both the present definition of length** and its possible revision which fixes the speed of light***. More significantly, however, the optical reference was well connected to the spectrum of the simplest of atoms, namely hydrogen, thus providing a logically satisfying anchor for theoretical comparisons in other spectral regions. Improvement in the resulting γ-ray secondary standards amounted to approximately a factor of forty.

Muonic Atoms—A Puzzle of the Early Seventies

The presently best developed theoretical structure in physics is quantum electrodynamics (QED). To a remarkable extent, it provides for an accurate and satisfying description of the hydrogen atom. The theory, including interaction with the radiation field, not only accounts for details of the spectrum of hydrogen itself but also for the spectra of all one-electron systems for which the nuclear charge is not too large. The structure of QED calculations is tolerant not only of replacing the nucleus of hydrogen by other nuclei, but also of replacing the electron itself by any other light negatively charged particle which interacts only with the electric field of the nucleus (i.e., does not experience the nuclear force). One such particle is the negative muon, often designated $\mu^{(-)}$. Except for its approximately 200 fold greater mass and its instability (which is not at issue here), $\mu^{(-)}$ appears identical to the electron, $e^{(-)}$.

It was thus a matter of surprise and heightened interest when, in the early seventies, a systematic pattern of discrepancies between theory and experi-

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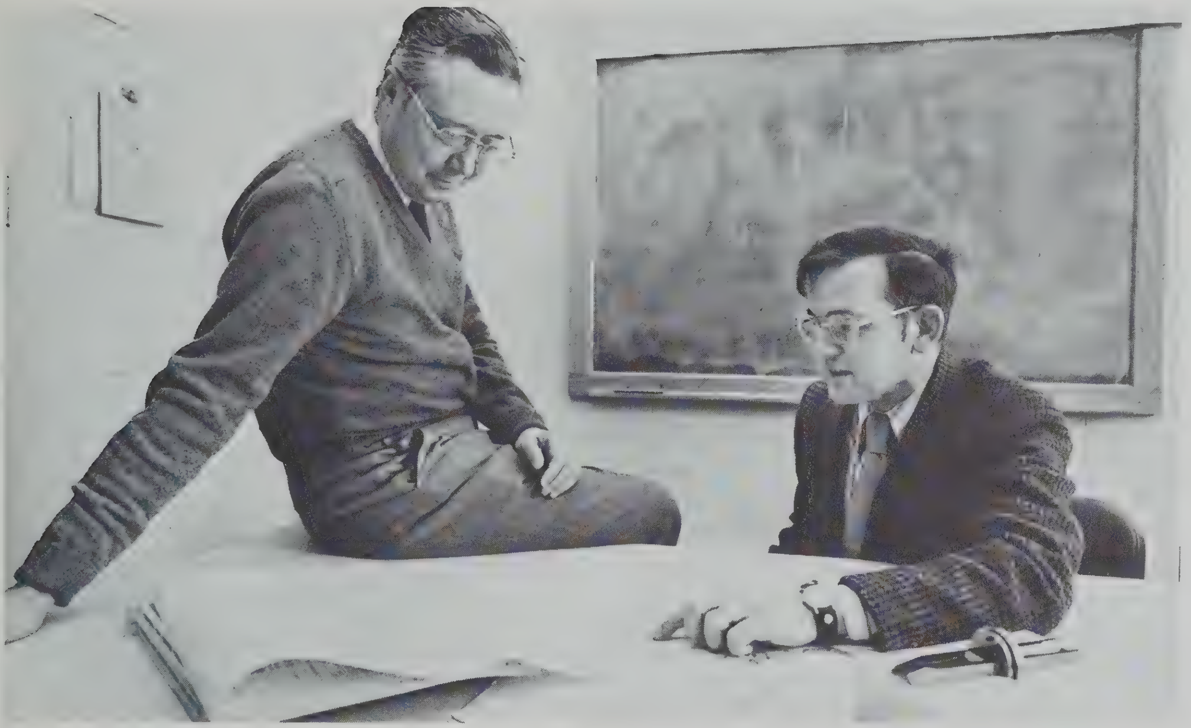
Deslattes is Senior Research Fellow and leader of the Quantum Metrology Group.

* *This constant defines the mole, the SI unit for amount of substance.*

** *At present, the 1960 krypton definition remains in effect.*

*** *A proposal to define length via setting c-constant has been recommended by the Consultative Committee for the Definition of the Meter.*



Richard Deslattes, group leader of the NBS Quantum Metrology Group, and his colleague, physicist Ernest Kessler, discuss data gathered in their experiments to improve the accuracy of gamma-ray wavelength measurements.

ment for muonic atom spectra began to appear. (The emission lines of these spectra are often called muonic x-rays because they appear in the x-ray region and extend into that of γ-rays). Through the mid-1970's, the pattern of discrepancies was elaborated on the experimental side while experiments having somewhat lesser precision claimed to see no discrepancy. Meanwhile, theorists restudied the QED calculations to see if improvement in calculational precision or more satisfactory treatment of corrections, e.g., vacuum polarization, might alter this discrepant picture. Such efforts, carried out within the conventional QED structure, were not successful, leading people to begin to speculate about a possible breakdown of QED or the need for a new elementary particle having the right set of interactions to mitigate observed differences between theory and experiment.

There are perhaps two not so obvious points that need noting: one is why such questions as the ones indicated above are provocative and the other is how such spectra are measured. Reasons for concern about this kind of conflict go beyond the obvious. Specifically, although the muon sees only the distribution of nuclear charge, the pi meson in a similar atomic situation is affected both by the presumably well known Coulomb force and by the less well known pion-nucleus interaction. Thus, in the case of pionic atoms, real knowledge of the pion-nucleus interaction could be gained provided the electrical effects could be subtracted. Validity of this subtraction depends totally on one's success at understanding muonic spectra where the strong force does not operate. In the absence of adequate understanding of muonic spectra, we are therefore inhibited from using pionic spectra to learn about the pion-nuclear interaction.

How these spectra have to be measured comes about in the following way: All atoms where either the central nucleus is replaced by something else or the orbiting electron is replaced by another something else can be called "exotic atoms." Among these exotic atoms, those involving pions, muons, and other heavier particles have spectra in the x-ray and γ-ray regions. Exotic atoms are never available in large numbers even at those massive accelerator facilities devoted to their production. Spectroscopy of these systems is therefore carried out using high efficiency spectroscopic systems which also record the position of one or more nearby γ-ray reference lines. Accuracy, therefore, in exotic atom spectroscopy is contingent on accuracy of the γ-ray reference lines used.

At the time when these discrepancies were being intensely studied, no one was focusing on the γ-ray reference lines as a possible source of some or all

of the difficulty. Indeed, the level of uncertainty in the reference lines did not differ markedly from the size of discrepancies observed. Thus, the time was ripe for a reevaluation of these reference standards which we have now provided.

The Optical to γ-Ray Connection

A significant disparity exists between the spectral scale of hydrogen (1 — 0.1 micrometer) and that of muonic lead (1 — 0.1 picometer). To bridge such a gap requires unusual effort since most simple measurement algorithms aim at the comparison of nearly equal magnitudes. A three-step process devised by the author and co-workers Ernest Kessler, Albert Henins and William Sauder, though complex, represents an appreciable simplification relative to alternative means for covering the 5-6 orders of magnitude over which precise relationships were required.

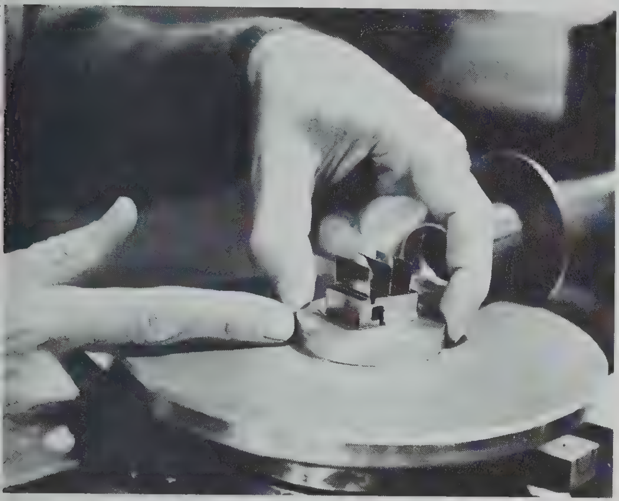
The three-step process involves first a measurement of the lattice period of a nearly perfect silicon crystal by means of combined x-ray and optical interferometry. In a second step, the silicon lattice measurement is disseminated to other specimens of silicon and germanium having sizes and shapes suited to x-ray and γ-ray diffraction investigations. In the third step, these "calibrated" specimens are used to diffract x-ray and γ-ray lines using instrumentation in which there is provision for accurate absolute angle measurement. This chain, then, connects the γ-ray and x-ray transitions studied directly with the optical reference laser and, thereby, the hydrogen atom. (See accompanying box for more detailed explanation of the apparatus.)

Each of the three steps was carried out with an accuracy of the order of 0.1 ppm. Since limitations of both theory and experiment in muonic atom spectra are at the level of several ppm, the accuracy of comparisons with theory is no longer limited by the quality of gamma ray reference values. When the changes in scale resulting from this work were combined with later and more carefully analyzed muonic x-ray measurements, the previously noted disagreements were found to be absent.

As the muonic atom question's resolution began to become clear, the group's attention turned to the situation of x-ray spectra from normal atoms. Although long ago these spectra too had been considered a testing grounds for QED, they are, in the author's opinion, much less sensitive in this regard than the muonic spectra. Since good theoretical atomic calculations are available and QED effects are calculable, what might be learned in a careful comparison between theory and experiment for normal atomic x-ray spectra?

The answer was not at first evident and remains somewhat obscure, even at present, but the empir-

Left and below. Physicist Albert Henins prepares to take an x-ray to test the quality of a monolithic silicon crystal interferometer.



ical situation is very clear indeed. When the rather few direct x-ray measurements already carried out by the NBS group were combined with several more results which had been obtained in Europe as ratios to γ-lines measured in the above described experiment, a limited but highly precise data set became available. Comparison of these data with atomic theory showed a striking systematic discrepancy between theory and experiment which varied linearly with atomic number (nuclear charge).

Thus, there is information of some theoretical significance to be gained by a systematic restudy of a broadly based sample of classical atomic x-ray spectra. New and more versatile machinery was required which would have a measure of portability, would enjoy the benefits of sophisticated real time computer control, and could also be used to work toward still higher γ-energies.

This major design and construction was undertaken by the author, Kessler, and Henins in collaboration with two visiting scientists, Ludo Jacobs from Leuven University in Belgium and Wolfgang Schwitz from Fribourg University in Switzerland. This new enterprise was completed in mid-1979 and is presently operating in the Radiation Physics Building, using the NBS 4 MeV electron Van de Graaff to excite characteristic x-ray spectra from principally heavy elements. ☐

A TECHNICAL LOOK AT THE INSTRUMENTATION

For the more technically interested, the following description and illustrations provide a closer look at the instruments used to improve the accuracy of γ-ray wavelength measurements.

Optical Measure of a Lattice Repeat Distance

The process of determining the 0.2 nm repeat distance in silicon in terms of the 632 nm stabilized laser is illustrated in figure 1. As noted in the text, the I_2 stabilized HeNe laser used is well connected to both current and future definitions of length as well as, more significantly, the spectrum of hydrogen or the Rydberg constant, R_∞. The separated crystal x-ray interferometer produces "fringes" as a function of the relative translation of the crystals along the illustrated direction (arrow). High reflectivity mirrors attached to each crystal element give a sharp optical fringe for each half period of the optical wavelength, approximately 316 nm. Evidently, 1648+f x-ray fringes pass by between each optical signal peak. The measurement strategy aimed at determining f in an efficient manner uses procedures reminiscent of the method of exact fractions in optics. After correction for wavefront curvature and path curvature, the result, stated at 25 °C for the particular vacuum float-zoned crystal used, was 192.017 07 picometers with a claimed accuracy of 0.1 ppm.

Crystal to Crystal Comparisons

For several practical reasons, the silicon crystal specimen used in the first step is unsuited for direct use especially in the case of γ-ray measurements. These require an assortment of other crystal types, especially germanium and other orientations both of silicon and of germanium. These comparisons are best made using the copious flux available from a normal x-ray source. The x-ray lines are, however, quite broad, having widths generally between 300 ppm and 1000 ppm. It would be quite difficult to attempt to use these in a direct way with any hope of reaching the 0.1 ppm level.

Instead, what is needed is some nearly null comparison scheme of the type shown in figure 2*. In this case, one can see that if the long, first crystal and the second crystal have the same spacing, the two reflections will occur at the same angle as the second crystal is slowly ro-

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* *Schemes of this type were first proposed by M. Hart, presently of King's College, London.*

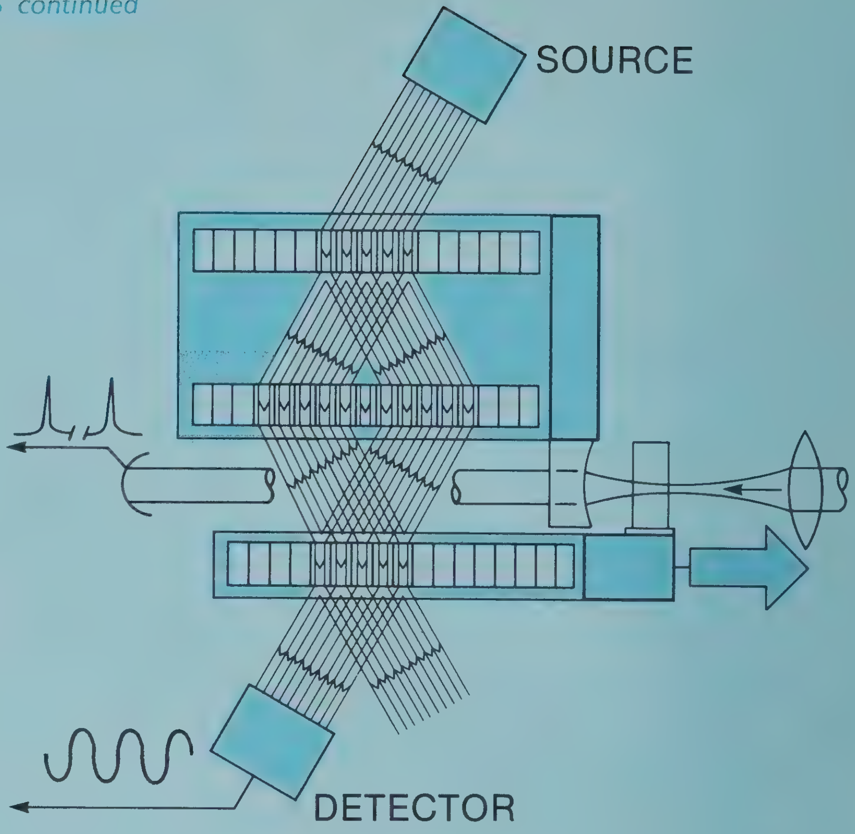

Figure 1—*Principle of x-ray/optical interferometer measurement of the (220) spacing in a silicon crystal. X-radiation from the Source at the top of figure is diffracted by the three crystal lamellas indicated. Radiation reaching the detector at bottom is modulated by the motion of the third crystal through the stationary wavefield produced by the first two. Laser radiation enters from the right passing through the optical interferometer whose mirrors as indicated are attached to the separated crystals. The occasional optical maxima suggested at left occurs at intervals of 316 nm while the x-ray lattice periods are 0.19 nm.*

tated. This occurrence will evidently be independent of x-ray wavelength for the case of exact equality. In the case that the two crystals differ slightly, the reflections will be displaced slightly by an amount proportional to the difference in spacings multiplied by the wavelength. For small differences, the effect of lack of wavelength precision has an effect which is diminished by a factor $\delta d/d$. Thus, x-ray line characteristics are no longer a dominant limitation.

In practice, the first crystal is not used directly as a reference since it might be bent or have a non-uniform spacing over the required large length. Instead, "standard" and specimen crystals are alternately placed in the second crystal position and the differences noted, thereby suppressing any influence of the first crystal. These procedures are clear for the case of equivalent reflections in the same crystal type. Going from one kind of crystal to another, for example, from silicon to germanium, requires matching some interplanar spacing in one species with another having different indices. This bit of gamesmanship works out favorably in many cases but nowhere more nicely than in the germanium to silicon comparison where there is an [800] reflection in germanium differing only by 200 ppm from the [355] reflection in silicon.

Using procedures such as those just described, it was possible to prepare several sets of crystals whose spacings were known almost as well as that of the original piece of silicon from which the interferometer specimen was extracted.

The γ-ray and x-ray measurements

In the third and last step, pairs of calibrated crystals were arranged in a transmission two-crystal instrument as shown in figure 3. In this case, the angular interval between the two diffraction conditions shown for wavelength, λ, is twice the Bragg angle, θ, where $\lambda=2d \sin \theta$ is exact for radiation travelling parallel to the plane of the figure. The value of d is available from the previous step so that information on λ is available to the extent that 2θ can be measured with the needed accuracy. There are several problems associated with carrying out such a measurement with targeted accuracy below the ppm level.

Crystal perfection has to be of such an order that the theoretically expected small diffraction widths are found at the small Bragg angles through which short wavelength γ-rays diffract from strong crystalline reflections. This has indeed been the case with diffraction widths at or near the 0.1 arcsecond being found for Bragg angles of the order of 1 degree. Typical linewidths are in the range 10-50 ppm, making it relatively simple to locate peak positions with the needed refinement provided that angle measurement systems are available with the needed sensitivity (~ below 1 milliarcsecond) and that these are calibrated relative to the cyclic angle of 2π radians or 360°

The required sensitivity was obtained by yet another application of interferometry. In this case, rather than for distance determination, in-

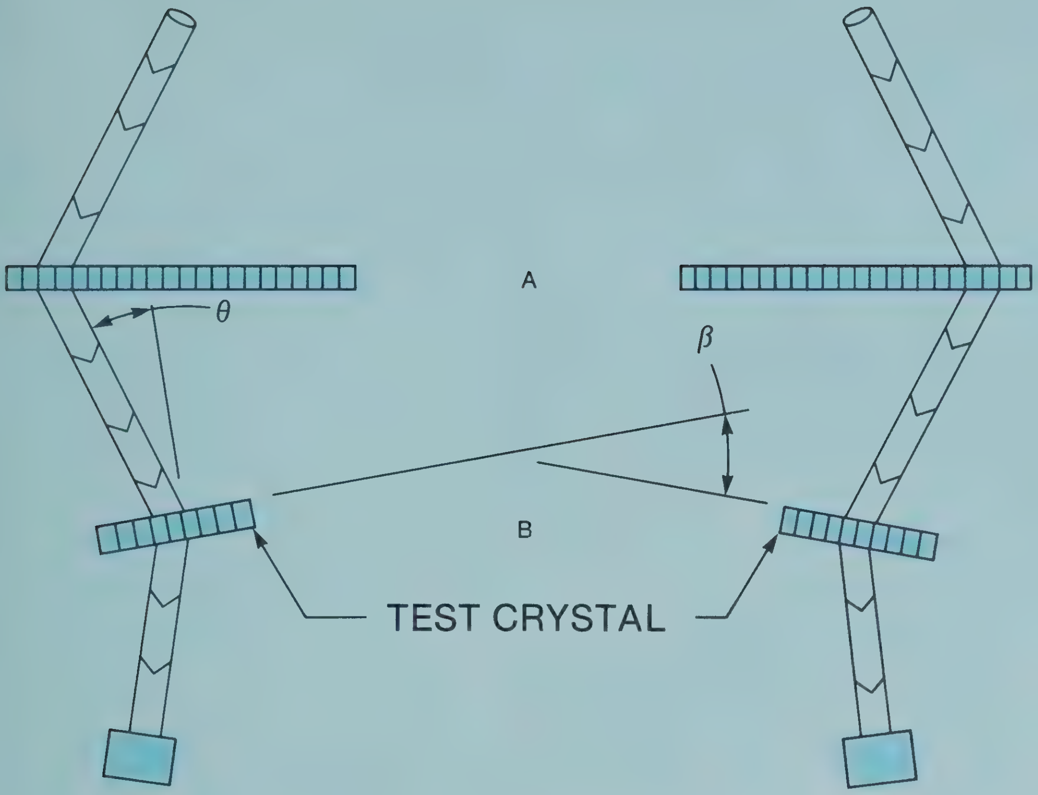

Figure 2—*A non-dispersive lattice transfer measurement. The symmetric paths indicated provide for diffraction angle differences proportional to lattice spacing differences with a wavelength dependence linear in the difference.*

terferometry was applied to angle measurement. In another departure, the fringe or intensity signals were replaced by polarization encoding. That is to say, the interferometer's output is a rotating polarization azimuth which traverses 180° rotation for each quadrant of phase shift. One of the current designs for such an interferometer is shown in figure 4. By choice of interferometer scale and beam folding, it was arranged that 90° polarization rotation corresponded to 70 milliarcsec of table rotation. Since polarization azimuth can be quite sensitively marked by extinction in a polarizing prism, measurement and control were readily available near the 0.1 milliarcsec level. This level of angle measurement and control is clearly adequate to the tasks undertaken. What remains is the question of absolute accuracy or calibration of the angle interferometer's scale.

Absolute angular measure differs from that of almost all other dimensioned quantities in that it needs no external definition and, hence, no external standards. Indeed, all that is needed is an arbitrary convention for some measure of subdivision within a closed circle to establish a common language. All such measures divide the closed circle or cyclic angle and no other reference is needed. Realization of such a first principles interferometer calibration requires some special steps since the angular ranges of the NBS instruments are always small. In the first one, the range was ± 2.5° while, in the latest version, this has been extended to ± 15°. In either case,

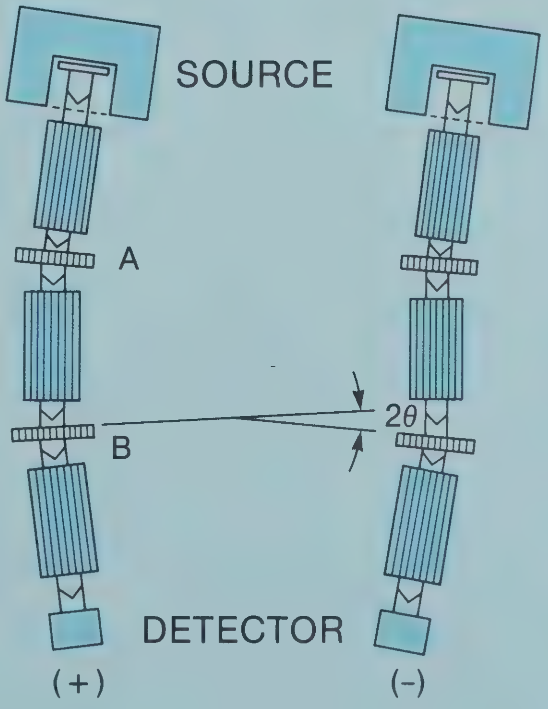

Figure 3—*Diagram of the symmetric transmission two-crystal spectrometer used for short wavelength x-ray and γ-ray measurements. Angle interferometry is used to carry out the needed accurate small angle measurements.*

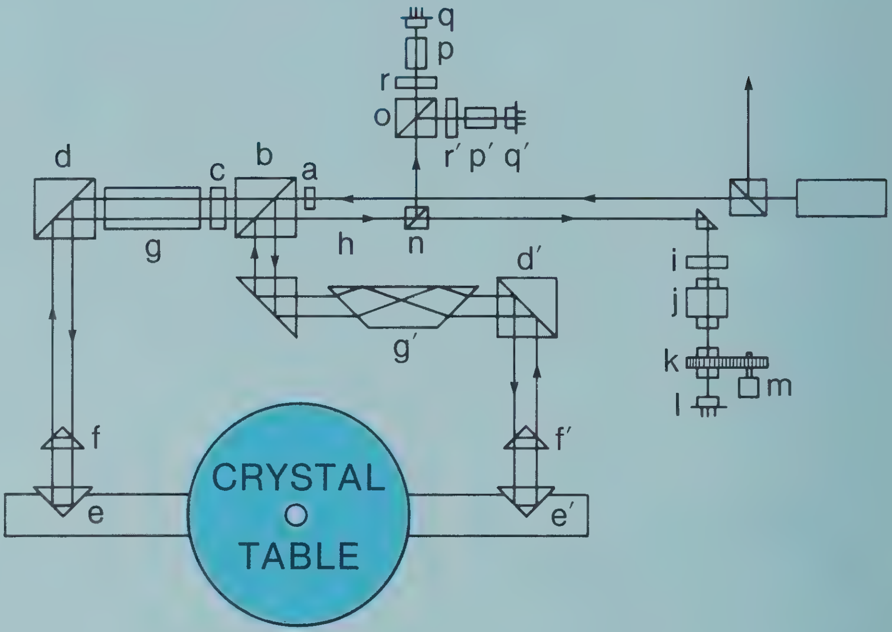

Figure 4—*A schematic illustration of one of the angle interferometers in current use. The optical details provided for a polarization output which rotates through 90° when the crystal table turns through approximately 0.07 arcsec. The plane of polarization is located precisely by the modulated null system, i, j, k, l, m. Larger motions of the table are noted by a counter and the optical system o, p, q, r. The main optical paths are determined by the polarizing beamsplitters b, d, d′, cube corner reflectors, e, e′ and roof prisms f, f′.*

the range is small compared to 360° and an intermediate step is required. This has been achieved in each case by using the angle interferometers to determine relative values of all external interfacial angles of solid quartz polygons. For the small angle instrument, a 72-sided polygon was used while, for the larger range instrument, a 24-sided object sufficed. With relative angle measures from interferometry, the closure condition fixes the sum of all angles to 360° thereby providing for absolute calibration of the interferometers. Such calibrations have been carried out over several years for the first instrument and have recently begun for the second. It is found that while an individual calibration run establishes the scale to a few times 0.01 ppm, at least the first instrument undergoes secular changes of the order of a few times 0.1 ppm per year. It was thus necessary to establish a calibration history and extract from it the particular calibration scale appropriate to γ-ray or x-ray measurements taking place at a time within this slowly changing situation. Errors associated with both the individual calibration exercises and the process of establishing an interpolation scale between them remain near 0.1 ppm in their effect.

Using the three-step process described above, some 24 γ-ray reference lines in the region $0.06 < E < 1.1$ MeV have now been remeasured with accuracies below 1 ppm. These results differ from previous ones both in respect to the improved accuracy levels and in providing a significant shift in average value. As an example, the 411 keV gamma ray from the ^{198}Hg daughter of ^{198}Au was previously estimated to have an energy of 411 794 eV with an uncertainty of 17 ppm. The NBS effort* gave 411 804.41 eV with an estimated uncertainty of 0.3 ppm. Similar changes have been found in other γ-ray lines, suggesting that previous measurements have generally been associated with a scale problem. At least in the case of the 411 keV line, this has been understood in terms of changes in estimates of the fundamental constants and in estimates of a principal x-ray line from uranium.

The above described γ-ray measurements lead to increased harmony between theory and experiment in the case of muonic spectra. Other measurements by the NBS group have focused on a few characteristic x-ray lines which had been involved in earlier definitions of the x-ray scale and contributed to those spectroscopic data by means of which systematic discrepancies have been found to persist over appreciable ranges of nuclear charge. While these new data have had a noticeable effect on the theory-experiment confrontation, they suggest, to a still greater extent, the potential utility of a more extensive and systematic study of these disagreements as functions of radial and angular quantum numbers as well as of nuclear charge.

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* *E. G. Kessler, Jr., R. D. Deslattes, A. Henins, and W. C. Sauder, "Redetermination of ^{198}Au and ^{192}Ir γ-Ray Standards between 0.1 and 1.0 MeV,"* Physical Review Letters, *Volume 40, 16 January 1978, page 171.*

NBS OPENS NEW INDUSTRIAL FURNACE FACILITY

by Michael Baum

The National Bureau of Standards has established a new experimental industrial furnace facility that will be used to study computerized control and instrumentation techniques for combustion systems.

According to the NBS project leader, Dr. Hratch Semerjian, the new furnace test facility will have three general objectives: to study automatic control systems that can improve the combustion efficiency of furnaces; to study the combustion characteristics of various fuels, especially synthetic and shale-derived fuels; and to evaluate diagnostic test methods for furnaces, boilers, and other combustion systems.

Initially, said Semerjian, the main emphasis at the furnace test facility will be on the development of control systems. "Part of the control problem that we're trying to address is that associated with the use of recuperators, which more and more people are using as fuel costs continue to climb," he said.

Recuperators are heat exchange systems that use the hot exhaust gases from the furnace to preheat the air going into the furnace, thus improving the overall efficiency of the system, explained Semerjian. When recuperators are used on furnaces with automatic control systems that are supposed to keep the air and fuel flows at optimum levels, they set up a complex control problem. The efficiency of the recuperator depends on the temperature of the furnace, which is determined in part by the temperature of the incoming air—which in turn depends on the temperature of the recuperator. Consequently, the use of the heat exchanger sets up a mechanical feedback system which must be carefully balanced for maximum efficiency.

Project leader Hratch Semerjian is shown at the recently established NBS experimental industrial furnace facility. The installation will be used to study computerized control and instrumentation techniques for combustion systems. Ceramic heat recovery system is visible above the far end of the furnace.

Moreover, any sudden change in the furnace, such as the addition of a metal ingot for melting, upsets the balance of the system. An automatic control system must be able to respond rapidly and effectively to such changes. Because recuperators are just now coming into widespread use, said Semerjian, automatic control procedures for dealing with this problem have not been developed.

The NBS test facility is unique in its use of a recuperator and extensive automated instrumentation. The laboratory is built around a modified slot forging furnace rated at 1 million Btu/hour (300 kW) and a standard ceramic industrial heat recovery system that can preheat air up to 850 °C. The instrumentation on the furnace, which includes temperature and pressure sensors, flow meters, and gas analysis equipment, is linked to a minicomputer which forms the basis of a computerized data acquisition system.

The researchers in the NBS Center for Mechanical Engineering and Process Technology are now developing a prototype computer program which will allow the system to run the furnace under automatic control. This work is expected to be completed within two years.

Currently the furnace burns either natural gas or number 2 fuel oil. Future plans include the addition of a "variable geometry" burner that will allow researchers to burn a variety of fuels in the furnace—coal-derived gaseous and liquid fuels and fuels derived from shale and tar sands, for example—and study their combustion characteristics. The gas analysis system for the test facility includes on-line analyzers for carbon monoxide, carbon dioxide, hydrocarbons, and various oxides of nitrogen and sulfur, and an off-line chromatograph.

Plans for future research, according to Semerjian, include using the furnace to test experimental non-intrusive instrumentation systems such as laser anemometers and interferometers for determination of flame characteristics, particulate size, and soot formation. Other optical emission/absorption diagnostics methods could conceivably provide separate information on every burner in a multiple-burner furnace, a serious limitation in most furnace stack instrumentation, according to Semerjian.

Baum is a writer and public information specialist in the NBS Public Information Division.

STAFF REPORTS

Surface Magnetism, page 26
Definitive Measurement, page 28

SURFACE MAGNETISM STUDIED USING POLARIZED ELECTRONS

NBS researchers have developed a new analytical tool for the study of surface magnetization. The technique involves the use of a special electron gun, similar to those used in low-energy electron diffraction (LEED), except that the electrons are spin polarized and the spin direction is modulated. This allows researchers to study spin-dependent scattering effects in the first few atomic layers of a single crystal surface.

Daniel T. Pierce, Robert J. Celotta, and Gwo-Ching Wang, Radiation Physics Division, B206 Metrology Building, 301/921-2053 and 2051.

At the surface of a magnetic crystal, the lack of translational invariance perpendicular to the surface and the reduced number of neighbors of the surface atoms cause the magnetic properties to be different from those in the bulk. In collaboration with Sam D. Bader and Gian P. Felcher of Argonne National Laboratory, we have developed a new way[1] to measure the magnetization of the outer few atomic layers of a single crystal surface based on polarized low-energy electron diffraction (PLEED). Due to the exchange interaction, the diffracted intensities depend on the relative orientation of the incident electron spin and the magnetization of the surface. The surface magnetization, its temperature dependence, and the transition temperature at the surface have been described by a number of theories which we now have a straightforward way to test experimentally. The increasing interest in lower dimensional magnetism and surface critical phenomena makes the development of our "surface magnetometer" especially timely. We also expect the surface magnetometer to have technological applications. In the push to further miniaturize magnetic devices by using thinner layers in magnetic storage media, the magnetic properties of the outer few layers will become very important. Our technique provides a research tool to study, for example, the magnetic changes at the surface of a recording head due to corrosion. Magnetic surfaces are also important in hydrogen storage and catalysis, such as in the coal gasification process. The surface magnetometer will allow the study of the role of the magnetic electrons in chemisorption on well characterized single crystal surfaces.

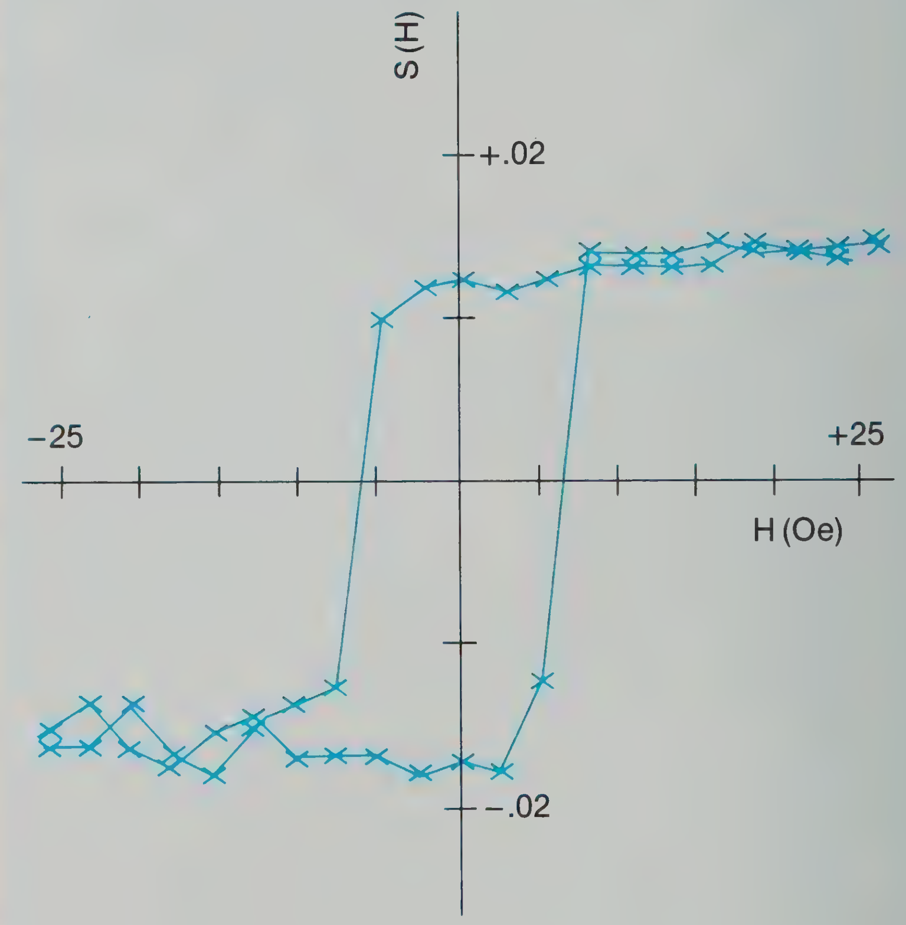

Figure 1—*A hysteresis curve, S(H), at an incident beam energy E=125 eV and angle of incidence θ_0=12°. The raw data points, which are connected by straight lines, were obtained between the S(T) measurements (× and +) of figure 2.*

Electrons are widely used in surface analytical techniques (such as photoemission, LEED, Auger spectroscopy), since due to their strong interaction with the electrons in the material, they probe only the outer few atomic layers. For example, in the Ni(110) surface which we studied, electrons with energy of about 100 eV can on the average penetrate and return through only two atomic layers without losing energy and being removed from the elastically scattered beam. Polarized electron scattering provides a way to study the surface of a well defined single crystal. The polarized electron probe is nondestructive, and has potentially very high spatial resolution although we have not exploited it thus far. One can envision a magnetometer for surface microstructure which could display magnetic domains and bubbles.

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[1] *R. J. Celotta, D. T. Pierce, G.-C. Wang, S. D. Bader, and G. P. Felcher, Physical Review Letters 43, 728-731 (1979).*

The measurement of surface magnetization by polarized electron scattering is made possible by the polarized electron gun which we have developed at NBS. The electron gun, which is based on photoemission from negative electron affinity GaAs, provides a constant intensity electron beam like an ordinary LEED gun, but the spin direction is modulated at a rapid rate. Since the incident electron beam intensity on the target crystal is constant, any a-c component of the scattered intensity synchronous with the polarization modulation must result from a spin-dependent scattering. The scattered intensity for incident electron spin parallel to the net spin in the ferromagnetic surface $I\uparrow\uparrow$ is in general different from the scattered intensity where the spins are antiparallel $I\uparrow\downarrow$. We measure a quantity S, which is this difference normalized to the spin averaged scattered intensity,

$$S = \frac{I\uparrow\uparrow - I\uparrow\downarrow}{I\uparrow\uparrow + I\uparrow\downarrow}.$$

We also correct for the fact that the incident polarization is 43 percent rather than 100 percent.

Theoretically, S depends on the exchange potential $J(E,\theta)$ and the Coulomb potential $V(E,\theta)$ seen by the electron scattering at an energy E and an angle θ. It

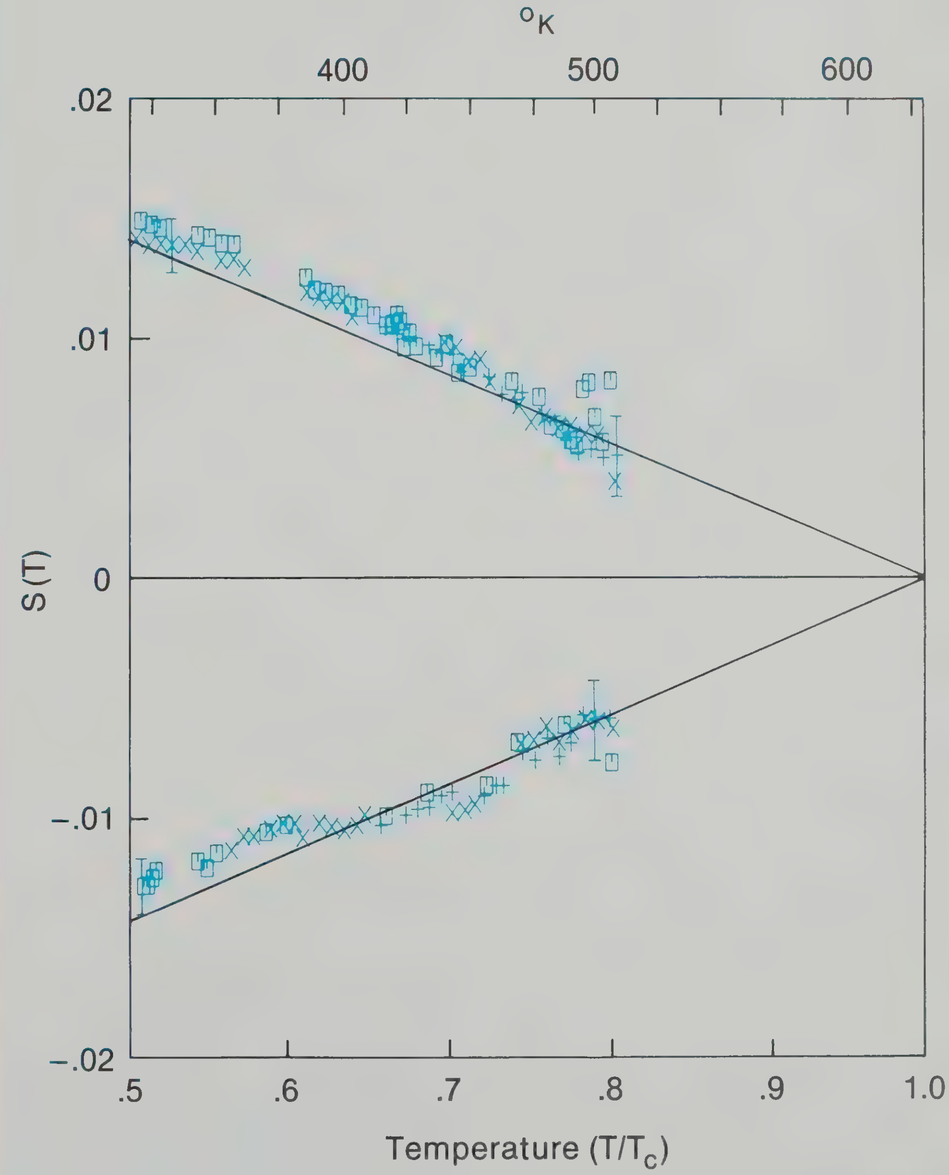


Figure 2—*The temperature dependence, S(T), at $E = 125$ eV and $\theta_0 = 12°$ is shown in the range $0.5 < T/T_c < 0.8$. There is less curvature than in the bulk magnetization in this temperature range because of the surface sensitivity. The different points denote three different cooling curves for each applied field direction. The applied fields were (squares) 18 0e, (pluses) 21 0e, (crosses) 26 0e. The estimated uncertainty is indicated by error bars at representative points. The straight lines represent a linear dependence of the magnetization to T_c (see text).*

can be shown in the first Born approximation that S is proportional to the average magnetization M of the outer few layers of the surface. If multiple scattering is included there may be higher order terms proportional to M^n, where n is odd, but in Ni these are very small and may be neglected.

The apparatus[2] consists of the polarized electron gun attached to a conventional surface analysis chamber. We cleaned a Ni(110) crystal by successive Ar ion bombardment and annealing cycles. The surface cleanliness was checked by Auger spectroscopy using a cylindrical mirror analyzer. Generally, it is not straightforward to scatter low energy electrons from the surface of a ferromagnet because of the deflection of the electrons in stray magnetic fields. These fields have been minimized by making the Ni crystal the "keeper" of the small electromagnet which applies the magnetic field that aligns the domains in the crystal.

We can measure S as a function of magnetic field to obtain a surface hysteresis curve S(H). Such a curve is shown in figure 1 for specular scattering at an angle of incidence of 12° and an energy of 125 eV. The maximum S in figure 1 is less than 0.02; this is a small signal but the noise level in this sensitive measurement technique is less than 0.001. Reversal of the magnetic field allows us to isolate any possible non-magnetic contribution to the spin dependent scattering. We emphasize that figure 1 is a hysteresis curve representing the magnetization of the outer two atomic layers. Such a curve is very sensitive to surface contamination. In particular, segregation of sulfur to the surface decreases the magnetization to zero. Future measurements will study the effects of adsorbates on the surface magnetization. We will investigate the existence of magnetic dead layers at the surface (considered unlikely from these first measurements) and the role of the magnetic d electrons in the bonding of adsorbed atoms.

One of many interesting questions is: How does the temperature dependence of the surface magnetization compare with that of the bulk? There are definite predictions from theoretical scaling laws relating the surface and bulk behavior in the critical region near the Curie temperature T_c. We were able to measure S(T) from 0.5 to 0.8 T_c and found that the temperature dependence of the surface magnetization shows much less curvature than that of the bulk; in fact, it is approximately linear. Measurements were made by heating above T_c and cooling in applied magnetic fields of 18, 21, and 26 Oe as represented by the different points in figure 2. The cooling was too fast to allow measurement near T_c. In future measurements we will have crystal temperature control and will measure up to T_c, thereby providing a test of theories of surface critical phenomena. We plan also to extend the measurements to low temperature in order to normalize S(T) to the saturation magnetization and to study the influence of surface spin waves.

In summary, this powerful new technique may be compared to neutron scattering which has been so successful in measuring bulk magnetic properties. We expect that analogous information will be obtained about surface magnetic properties using our new PLEED technique.

DEFINITIVE MEASUREMENT OF CONSTITUENTS IN HUMAN SERUM

Researchers in the NBS Center for Analytical Chemistry are currently developing definitive methods for measuring the organic constituents of human serum, specifically cholesterol, glucose, uric acid, urea, and creatinine. A definitive method is designed to generate results of high precision that are essentially free of bias, and as such, will be the most accurate method known for that constituent. The goal of the researchers is to demonstrate that, for a series of independent samples taken from a stable serum pool over a period of time, analyses using the definitive method will produce information with a relative standard deviation of less than 1 percent.

Edward White V, Michael J. Welch, Robert Schaffer, Harry S. Hertz, Organic Analytical Research Division, A113 Chemistry Building, 301/921-2153.*

In clinical chemistry, the need exists for precise, controllable, yet simple reference methods. Such methods should provide "true" values and, therefore, the accuracy of the reference method must be known. A definitive method gives analyte values for serum pools against which values obtained using a candidate reference method can be judged. Definitive methods have other applications; for example, the College of American Pathologists uses them to determine how well individual laboratories perform their routine tests. The definitive methods we are developing will also be used in the certification of constituent concentrations in a proposed human serum Standard Reference Material (SRM). This material should be of particular interest to manufacturers of clinical instruments and supplies since the certified analyte concentrations will be known to a high degree of certainty.

In this research, we are using isotope dilution mass spectrometry (IDMS) for the definitive method measurements. The technique involves the addition of an isotopically labeled analog of the compound of interest to the sample to act as an internal standard. The isotope that is added is a stable, nonradioactive (but in nature less abundant) one that is substituted for the more abundant isotope of that element in the molecule of interest. For example, deuterium may be substituted for hydrogen, or carbon-13 can be substituted for carbon-12. These substitutions have little or no effect on the chemical or physical properties of the molecule.

Thus, if a labeled form of the compound of interest is added to a serum sample and the sample is processed to isolate some of the mixture of both forms of the compound, the ratio of labeled to unlabeled compound, should remain constant. Since these two forms differ in molecular weight, they can be differentiated by mass spectrometry.

---

[2] *G.-C. Wang, B. I. Dunlop, R. J. Celotta, and D. T. Pierce, Physical Review Letters 42, 1349-1352 (1979).*

** College of American Pathologists research associate at NBS.*

In the mass spectrometer, molecules are ionized and also fragmented to some extent based on the characteristics of the molecule itself as well as the means of ionization. The ions that form are accelerated into a magnetic field that is set to allow only ions of a given mass (or more correctly a given mass to charge ratio) to reach the detector at any given time. For our isotope dilution measurements, the magnetic field is systematically switched between a selected mass of interest from the unlabeled compound and one from the labeled compound.

For each 1-second time period that the magnetic field is fixed on a selected mass, the detector signal is measured and converted to a digital count, which is proportional to the number of molecules of the compound. Our data system accumulates the individual 1-second counts separately for the two masses and calculates a ratio between the total counts at each mass. Since the sample generally enters the mass spectrometer as a peak from the gas chromatograph, the data system corrects the accumulated counts at each mass for the background measured just prior to its entry into the mass spectrometer.

The entire IDMS procedure generally involves the following steps:

(1) addition of a measured quantity of the labeled material to a known quantity of serum;

(2) thorough mixing and equilibration to ensure sample homogeneity;

(3) lyophilization and other steps to isolate the labeled and unlabeled compound from the serum matrix;

(4) derivatization, if necessary, to convert the compound to a form more suitable for analysis;

(5) injection into a gas chromatograph for a final separation of the compound from impurities;

(6) elution of the compound from the gas chromatograph into the mass spectrometer and generation of mass spectrometric data in the form of a ratio of the labeled to unlabeled molecules in the sample; and

(7) data reduction to find the concentration of unlabeled compound originally in the serum.

Simply measuring the samples in this manner is not sufficient to generate highly accurate and precise results. Calibration standards, consisting of mixtures of known amounts of high purity unlabeled material (NBS SRM's are used) and the labeled material must be run frequently. Our technique involves bracketing each sample both in time and isotopic ratio with calibration standards. That is, immediately preceding the measurement of a sample, a calibration standard with a ratio slightly lower (or higher) than the sample is measured. And then immediately following the measurement of the sample, another calibration standard, whose ratio is slightly higher (or lower) than the sample, is also measured. The quantity of analyte in the sample is calculated by linear interpolation. To ensure that we do not have a bias introduced by the order of analysis, the order of measurement is reversed on a second day. The two days' values are averaged to produce the reported result.

Although our procedure has given excellent results, there are pitfalls of which one must be aware. Each new analyte presents unique problems. These may be associated with the separation of the analyte from potential interferences, its derivatization into a form suitable for gas chromatography, or its mass spectrometric behavior. In addition, the mass spectrometer itself presents problems for this type of analysis. Small aberrations that would not affect the normal uses of a mass spectrometer can noticeably worsen the precision of this type of work and must be removed or corrected.

(Four Pairs of Samples Prepared at 1-Week Intervals).

Serum Pool	Mean m mol/L	Relative Standard Deviation (%)
A	3.4298	0.33
B	4.7190	0.30
C	6.1494	0.36
D	7.4556	0.31
E	8.7881	0.32

Table 1. Cholesterol Determinations in Serum by Isotope Dilution Mass Spectrometry.

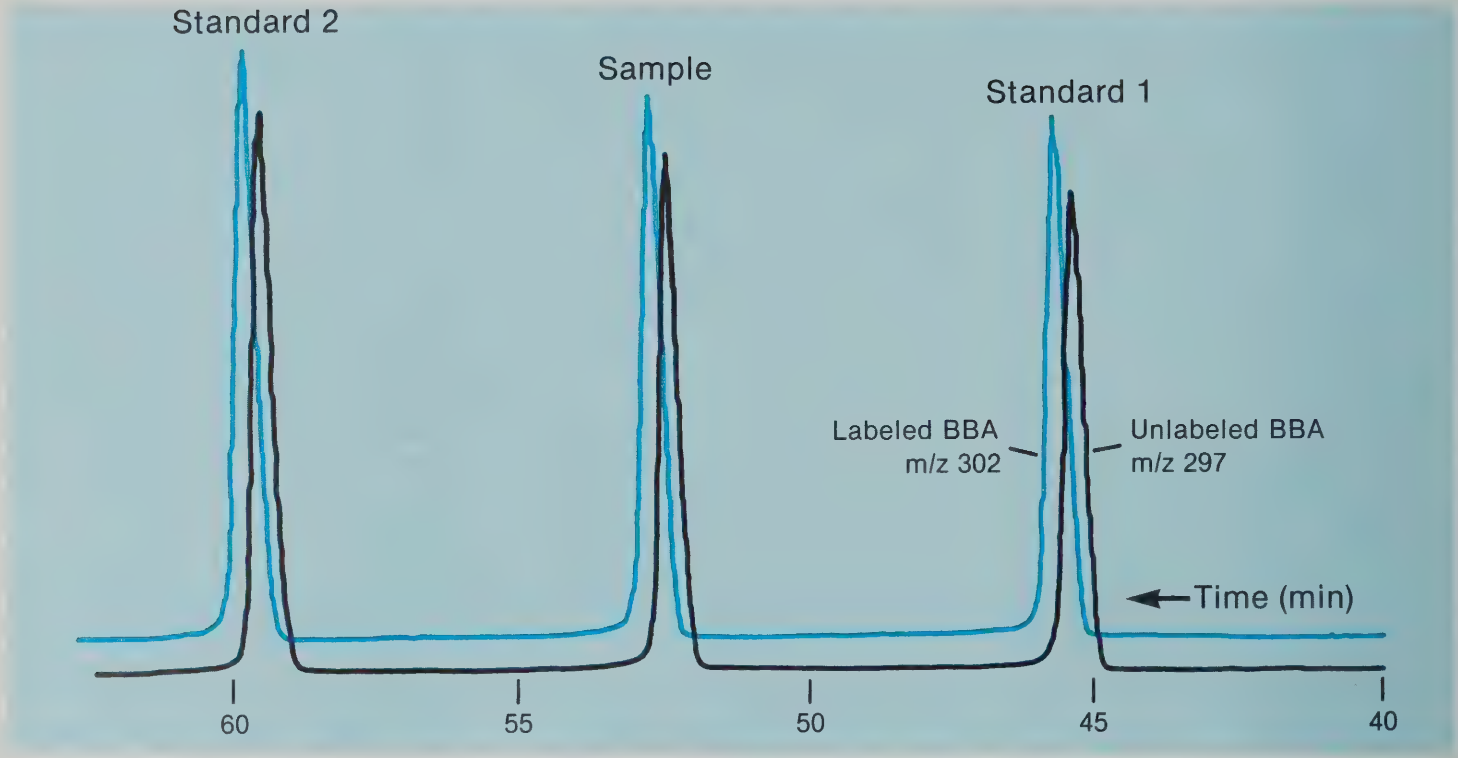
Cholesterol[1] is the first analyte for which a definitive method has been developed. The labeled material, cholesterol-d_7 which has seven deuteriums, is added to the serum. The serum is hydrolyzed to free all bound cholesterol. Then the mixture of labeled and unlabeled cholesterol is extracted and converted to the trimethylsilyl derivative. Samples are injected into a gas chromatograph and the derivative elutes directly into the mass spectrometer. Measurements are made in the electron impact mode at masses 458 (unlabeled) and 465 (labeled). The samples are measured as described above, along with calibration standards derivatized in the same manner.

Table 1 summarizes the results of IDMS analyses of five serum pools. The overall precision is more than adequate for the intended purpose. Remeasurement of several of these samples at other masses and by chemical ionization gave virtually identical results, indicating that there was no interference at either of the masses.

The accurate determination of serum glucose has long been a problem in clinical chemistry. We have developed two IDMS methods for glucose which produce results that agree within 1 percent of each other. Both methods utilize a carbon-13 labeled glucose for the internal standard.

The first method involves a condensation reaction of glucose with acetone to form 1,2:5,6 di-0-isopropylidene-D-glucofuranose, commonly known as diacetone glucose (DAG). The procedure requires three thin-layer chromatography steps to separate DAG from other monosaccharide-acetone condensation products which might interfere with the DAG measurements. The samples are analyzed by gas chromatography-mass spectrometry in a manner similar to that for cholesterol. Due to the decline in serum glucose levels with storage, demonstration of this method's reproducibility over long pe-

Figure 1—*An ion chromatogram showing the responses of labeled and unlabeled glucose BBA for a serum sample bracketed by standards.*



riods of time has not been possible. Because of this uncertainty about reproducibility over long periods, work was begun on a second method.

The second method involves an adaptation of a previously published IDMS method for serum glucose[2]. This method utilizes the reaction of glucose with butylboronic acid and acetic anhydride to form D-glucofuranose cyclic 1,2:3,5-bis (butylboronate) acetate or glucose BBA, for short. The wet chemistry in this procedure is much simpler than the DAG procedure, since the separation of the glucose BBA from other monosaccharide BBA derivatives is left to the gas chromatographic column. Whereas, cholesterol and DAG were separated on a packed column, a glass capillary column was required to obtain the necessary separation for glucose BBA. With capillary columns, the retention times are generally much longer (45 minutes for glucose BBA); thus our procedure had to be altered. By injecting one standard, followed 7 minutes later by a sample, and 14 minutes later by the other standard, we were able to maintain high precision, while saving time. An example of this is shown in Figure 1. We found no measurable interference of one injection with another.

When the results from the DAG and glucose BBA methods were compared for identical samples from three serum pools, both were found to exhibit excellent precision (CV's of 0.15 to 0.29 percent). However, the glucose levels found by the DAG method were 0.6 to 0.9 percent higher than the levels found by the BBA method. While these differences are small, they are larger than the uncertainty of our measurements. Remeasurement of some of these samples from the two methods under isobutane chemical ionization conditions gave results very similar to the original results, thus indicating that both methods are free of any significant interference in the measurement process. We feel the difference may be related to the amount of unlabeled glucose liberated from the serum protein by the two methods and this possibility is under investigation.

Work is nearly complete on a definitive method for uric acid in serum. The labeled material added is uric acid $^{15}N_2$ and the derivatization involves conversion into the tetraethyl derivative of which there are several isomers. Two isomers predominate; these are isolated from the matrix and from each other. Calibration standards are similarly derivatized and isolated in order that each isomer isolated from the serum can be measured by bracketing with the corresponding isomer from the standards.

Research is underway on suitable derivatives for urea and creatinine, the compounds for which we next hope to develop definitive methods.

Other NBS scientists involved in this research are: Alex Cohen, John Mandel, Stanley Meiselman, Robert Paule; Lorna T. Sniegoski, Tung Sun, and Pieter Van DerLijn.

References

1. *A. Cohen, H. S. Hertz, J. Mandel, R. C. Paule, R. Schaffer, L. Sniegoski, T. Sun, M. J. Welch, and E. White V, Total Serum Cholestrol by Isotope Dilution Mass Spectrometry. A Candidate Definitive Method; submitted to Clinical Chemistry.*

2. *D. Bier, W. Sherman, W. Holland, and D. Kipnis,* Proc. Int. Conf. Stable Isot. Chem., Biol., Med., 1st, 397 (1973).

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CONFERENCES

For general information on NBS conferences, contact JoAnn Lorden, NBS Public Information Division, Washington, D.C. 20234, 301/921-2721.

IMPLANT RETRIEVAL: MATERIAL AND BIOLOGICAL ANALYSIS CONFERENCE

A conference will be held May 1 and 2, 1980, at NBS in Gaithersburg, Maryland, to review the state-of-the-art in material and biological analysis associated with implant retrieval. The agenda will include consideration of uniform practices for obtaining and evaluating data concerning implant retrieval as well as discussions of advances in material and biological science relevant to implant retrieval.

Invited papers will be presented in the following areas in four half-day sessions: Bulk Phenomena—metal fatigue, fracture, deformation, creep, polymer degradation, biomechanics; Interfacial Phenomena—wear, corrosion, infection, fixation, biological response; Release Phenomena—response to wear debris and corrosion products, polymer migration, systematic effects, long-term effects; and Information Utilization—data center coordination, systems for retrieval/analysis, data utilization, legal and political barriers.

The conference is sponsored by NBS, the Bureau of Medical Devices of the Food and Drug Administration, the National Institutes of Handicapped Research, the Veterans Administration's Rehabilitative Engineering R&DS, and the American Society for Testing and Materials.

For further information, contact Dr. A. William Ruff, B118 Materials Building, 301/921-2811.

FIFTH INTERNATIONAL SYMPOSIUM ON ULTRASONIC IMAGING AND TISSUE CHARACTERIZATION

The state-of-the-art, new directions of technology, and research opportunities in ultrasonic imaging and characterization of tissues will be featured at a symposium to be held June 2-4, 1980, at NBS in Gaithersburg, Maryland. The meeting, sponsored by NBS and the National Institutes of Health, will provide physicians, engineers, physical scientists, and mathematicians a unique vehicle for sharing their contributions to this important developing area of medical diagnosis.

The scope of the symposium will embrace all aspects of ultrasonic diagnostics. Sessions will be devoted to 1) techniques for measurement of ultrasonic properties of tissue (velocity, attenuation, absorption, scattering, impedance); 2) imaging techniques, including transducer and array technology; 3) computerized tomography; 4) signal processing and pattern recognition; 5) Doppler techniques; 6) microscopy; 7) ultrasonic characteristics of tissue and their dependence on physical and biological variables (e.g., ultrasonic frequency, temperature); 8) equipment standardization and calibration; and 9) tissue phantoms.

The Symposium will couple with the Second International Symposium on Ultrasonic Materials Characterization to be held at NBS, June 4-6, 1980. (See description below). The one-day overlap between the two meetings will afford an opportunity for medical and materials researchers to interact and discuss common issues. These symposia are the only international meetings exclusively devoted to fundamental ultrasonic measurement methods that offer potential for practical assessment of material performance.

For further information, contact (general) Kathy Stang, B348 Materials Building, NBS, 301/921-3295, or (technical) Dr. Melvin Linzer, A366 Materials Building, NBS, 301/921-2611.

SECOND INTERNATIONAL SYMPOSIUM ON ULTRASONIC MATERIALS CHARACTERIZATION

The Second International Symposium on Ultrasonic Materials Characterization is being held to provide a forum for researchers to discuss the present state-of-the-art of ultrasonic measurement methods for characterizing materials and defects. New directions and research opportunities in these fields will be discussed and an opportunity will be provided for participants to compare progress in medical and industrial ultrasonics.

The meeting will serve to relate new ultrasonic measurement procedures to material parameters important to performance. The symposium will be held June 4-6, 1980, at NBS in Gaithersburg, Maryland.

Symposium topics will include ultrasonic methods for determination and verification of material properties (stress, hardness, grain and bonding, for example) by measurements of such parameters as velocity, attenuation, absorption, and scattering; characterization of defects in terms of type, size, shape, orientation, and location; ultrasonic instrumentation and signal processing for imaging, tomography, microscopy, pattern recognition, and sensitivity enhancement; transducer and array technology (including electromagnetic, polymer, and laser interaction); and ultrasonic standards and calibrations.

A Tutorial Session, open to registrants of both meetings, will be held on the afternoon of June 1, preceding the Tissue Characterization Symposium.

For further information, contact (general) Kathy Stang, B348 Materials Building, NBS, 301/921-3295, or (technical) Dr. Melvin Linzer, A366 Materials Building, NBS, 301/921-2611.

CONFERENCE CALENDAR

***April 14-15**

ADVANCES IN FIRE PHYSICS, A conference honoring P. H. Thomas, NBS, Gaithersburg, MD; sponsored by NBS; contact: James Quintiere, B356 Polymers Building, 301/921-3242.

April 16-18

CONFERENCE ON FIRE ENGINEERING, NBS, Gaithersburg, MD; sponsored by NBS; contact: Irwin Benjamin, B250 Polymers Building, 301/921-3255.

May 1-2

IMPLANT RETRIEVAL: MATERIAL AND BIOLOGICAL ANALYSIS, NBS, Gaithersburg, MD; sponsored by NBS, DOC, FDA, DHEW, VA, and ASTM; contact: A. W. Ruff, B118 Materials Building, 301/921-2966.

May 5-7

TOPICAL CONFERENCE ON BASIC OPTICAL PROPERTIES OF MATERIALS, NBS, Gaithersburg, MD; sponsored by NBS in cooperation with OSA; contact: Albert Feldman, A251 Materials Building, 301/921-2840.

***May 8-9**

TRACEABILITY FOR IONIZING RADIATION MEASUREMENTS, NBS, Gaithersburg, MD; sponsored by NBS; contact: H. T. Heaton, C229 Radiation Physics Building, 301/921-2551.

***May 13-15**

METROLOGY OF MODERN ELECTRONIC INSTRUMENTATION, NBS, Gaithersburg, MD; sponsored by NBS; contact: Barry A. Bell, B162 Metrology Building, 301/921-2727.

May 13-15

MEDILOG 80, NBS, Gaithersburg, MD; sponsored by NBS and DOD; contact: Charles Hulick, A740 Administration Building, 301/921-3465.

May 29

IEEE COMPUTER CONFERENCE, NBS, Gaithersburg, MD; sponsored by NBS and IEEE; contact: Frances Nielsen, B212 Technology Building, 301/921-2601.

June 2-6

6TH INTERNATIONAL CONFERENCE ON VACUUM ULTRAVIOLET RADIATION PHYSICS, UNIVERSITY OF VIRGINIA, CHARLOTTESVILLE, VA; sponsored by NBS, NRL, University of Virginia, NSF, DOE, IUPAP; contact: Robert Madden, A251 Physics Building, 301/921-2031.

June 2-4

FIFTH INTERNATIONAL SYMPOSIUM ON ULTRASONIC IMAGING AND TISSUE CHARACTERIZATION, NBS, Gaithersburg, MD; contact: Melvin Linzer, A366 Materials Building, 301/921-2611.

June 4-6

SECOND INTERNATIONAL SYMPOSIUM ON ULTRASONIC MATERIALS CHARACTERIZATION, NBS, Gaithersburg, MD; sponsored by NBS and ASNT; contact: Harold Berger, B312 Physics Building, 301/921-3331.

June 19

19TH ANNUAL TECHNICAL SYMPOSIUM PATHWAYS TO SYSTEM INTEGRITY, NBS, Gaithersburg, MD; sponsored by NBS and ACM; contact: Carol Wilson, A252 Technology Building, 301/921-3861.

***June 22-27**

NATIONAL CONFERENCE ON WEIGHTS AND MEASURES, Shoreham-Americana, Washington, D.C.; sponsored by NBS and NCWM; contact: Harold Wollin, A211 Metrology Building, 301/921-3677.

***June 23-25**

HIGH RESOLUTION INFRARED APPLICATIONS AND DEVELOPMENTS, NBS, Gaithersburg, MD; sponsored by NBS; contact: Jon Houghen, B268 Physics Building, 301/921-2021.

***July 22-24**

NBS EMI METROLOGY SEMINAR, NBS, Gaithersburg, MD; sponsored by NBS; contact: M. Gerald Arthur, EMI/Radiation Hazards Group, Electromagnetic Fields Division, NBS, Boulder, CO; 303/499-1000, ext. 3603.

***August 14-15**

ASME SYMPOSIUM ON CRITICAL MATERIALS AND FABRICATION ISSUES, St. Francis Hotel, San Francisco, CA; sponsored by NBS and ASME; contact: Jeffery Fong, A302 Administration Building, 301/921-2631.

***September 29 October 1**

CERAMICS AS ARCHEOLOGICAL MATERIAL, NBS, Gaithersburg, MD; sponsored by NBS and Smithsonian Institution; contact: Alan Franklin, A355 Materials Building, 301/921-2901.

***October 7-9**

COAL CONVERSION, NBS, Gaithersburg, MD; sponsored by NBS and DOE; contact: Samuel Schneider, B308 Materials Building, 301/921-2894.

***December 2-4**

TWELFTH ANNUAL PRECISION TIME AND TIME INTERVAL (PTTI) APPLICATIONS AND PLANNING MEETING, NBS, Gaithersburg, MD; sponsored by NBS, NASA and Agencies of the Department of Defense; contact: Arthur McCoubrey, A363 Physics Building, 301/921-3301.

** New Listings*

PROCEEDINGS ON THE CHARACTERIZATION OF HIGH TEMPERATURE VAPORS AND GASES

*Tenth Materials Research Symposium on Characterization of High Temperature Vapors and Gases, Nat. Bur. Stand. (U.S.) Spec. Publ. 561, Vol. 1 and 2, 895 pages (Sept. 1979) Stock No. 003-003-02124-5, $20.00 (sold in sets only).**

A comprehensive collection of state-of-the-art papers on the characterization of high temperature vapors and gases has been published by the National Bureau of Standards. The two-volume, 895-page report includes 65 papers presented at the 10th annual Materials Research Symposium held at NBS headquarters in Gaithersburg, Maryland, September 18-22, 1978, as well as edited transcripts of question and answer sessions.

In addition, 12 papers describing important new or improved techniques which were not presented during the symposium are included to make this a more complete report of the major new research in this field.

Researchers concerned both with the basic properties of hot vapors and gases and the applied problems of the extremes of temperature, pressure, and chemical activity should find these volumes of particular interest.

Modern technology increasingly requires materials and processes to function at high temperatures, making detailed knowledge of vapor, gaseous and plasma phases of chemicals more important. For example, the future success of new energy technologies such as coal-fired magnetohydrodynamic (MHD) generators, coal gasification, and nuclear fusion depends largely on materials performance in hot, chemically reactive, gaseous atmospheres.

At the NBS symposium, for the first time, an assessment of the various techniques of characterizing these atmospheres —both experimental and theoretical— was attempted by a large group of specialists in the field.

Attending the symposium were internationally recognized experts in the measurement science and technology of high temperature vapors, gases, flames, and, to a lesser extent, plasmas. Key foreign laboratories including those of the U.S.S.R., France, England, Japan, West Germany, Canada, and Mexico contributed to the proceedings. Academic, industrial, and government institutions from the United States made substantial contributions.

Throughout the text the authors have made an effort to cite useful follow-up references, and the editor has listed additional major reference sources representing the past decade of research in this and related fields.

The proceedings are grouped under ten major headings including:

Volume One

—Classical vaporization techniques

—Mass spectrometric techniques—Knudsen effusion

—Mass spectrometric techniques—high pressure sampling

—Molecular structure determination—matrix isolated species

—Molecular structure determination—vapor species

Volume Two

—Spectroscopic diagnostic techniques—species concentration

—Spectroscopic diagnostic techniques and temperature measurement

—Rate processes in gases—homogeneous systems

—Rate processes in gases—heterogeneous systems

—Thermodynamic data and applications

NEUTRON RADIATION FROM MEDICAL ACCELERATORS

Proceedings of a Conference on Neutrons from Electron Medical Accelerators, Nat. Bur. Stand. (U.S.), Spec. Publ. 554, 172 pages (Sept. 1979) Stock No. 003-003-02115-6, $4.75.

Problems related to the neutron radiation produced by high-energy electron accelerators often used to treat cancer patients are reviewed in a collection of 18 papers published by the National Bureau of Standards.

The papers were presented originally at an April 1979 two-day conference on the subject sponsored jointly by NBS and the Bureau of Radiological Health (BRH) with cooperation from the American Association of Physicists in Medicine (AAPM).

When these medical electron accelerators are operated at energies above about 10 MeV (million electron-volts), neutrons can be produced by photonuclear reactions within the target used to produce the desired x-rays or by the x-ray beam interacting with the beam-defining collimators.

One of the chief difficulties in measuring the neutron radiation is that the neutrons are only a small fraction of the number of x-rays in the treatment beam. The instruments usually used to measure neutrons respond equally well to the x-rays, hence special instruments and techniques must be used to measure the neutrons.

The biological effect of a small amount of absorbed neutron energy is equivalent to the effect of a much larger amount of absorbed x-ray energy, making this neutron contamination an important concern. Ideally, the total dose the patient receives during treatment should account for the dose due to the small neutron components.

The collimators which limit the x-ray beam will not limit the neutrons as effectively. Thus the neutron radiation delivered to the patient is received over a wide area. Other factors which should be

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OF THE NATIONAL BUREAU OF STANDARDS

considered are the number of neutrons produced by photonuclear interactions of the treatment x-ray beam within the body of the patient and determination of the dose due to the x-ray beam scattered within the patient by the body. Medical authorities and researchers are also interested in weighing the beneficial effects of using higher-energy beams in cancer treatments against unwanted side effects due to scattered x-rays and photonuclear-produced neutrons.

The 18 papers presented in this compilation discuss the uses of high-energy beams in radio-therapy; the existing regulations in this area and how they are interpreted and applied; the biological effects of neutron radiation and how the hazard associated with a given accelerator can be assessed; the instrumentation, measurement and calculation techniques available for determining the neutron radiation in primary and leakage beams from accelerators; and the shielding and design techniques that can be used to reduce the contamination to a minimum.

Atomic and Molecular Studies

Hoffman, J. D., Theory of Flow-Induced Fibril Formation in Polymer Solutions, J. Res. Nat. Bur. Stand. (U.S.), *84,* No. 5, 359-384 (Sept.-Oct. 1979).

Computer Science and Technology

Leong-Hong, B., Guidelines for Documentation of Computer Programs and Automated Data Systems for the Initiation Phase, Nat. Bur. Stand. (U.S.), Fed. Info. Process. Stand. Publ. (FIPS PUB) 64, 54 pages (Aug. 1979).

Health and Safety

Misra, D. N., Isoionic Isotope Exchange with Hydroxylapatite and the Dilution Effect, J. Res. Nat. Bur. Stand. (U.S.), *84,* No. 5, 395-406 (Sept.-Oct. 1979).

Velapoldi, R. A., Paule, R. C., Schaffer, R., Mandel, J., Murphy, T. J., and Gramlich, J. W., A Reference Method for the Determination of Chloride in Serum, Nat. Bur. Stand. (U.S.), Spec. Publ. 260-67, 97 pages (Nov. 1979) Stock No. 003-003-02136-9, $3.75.

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Arthur, M. G., Reeve, G. R., A Standard for RF Modulation Factor, Nat. Bur. Stand. (U.S.), Tech. Note 1106, 92 pages (Sept. 1979) Stock No. 003-02125-3, $3.50.

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Blackburn, D. L., Semiconductor Measurement Technology: An Automated Photovoltaic System for the Measurement of Resistivity Variations in High-Resistivity Circular Silicon Slices, Nat. Bur. Stand. (U.S.), Spec. Publ. 400-52, 41 pages (Nov. 1979) Stock No. 003-003-02138-5, $2.00.

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Mathematical and Statistical Methods

Domich, P., Lawrence, J., and Shier, D., Generators for Discrete Polynomial L_1 Approximation Problems, J. Res. Nat. Bur. Stand. (U.S.), *84,* No. 6, 455-488 (Nov.-Dec. 1979).

Leighton, F. T., A Graph Coloring Algorithm for Large Scheduling Problems, J. Res. Nat. Bur. Stand. (U.S.), *84,* No. 6, 489-506 (Nov.-Dec. 1979).

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American National Standard N538 Classification of Industrial Ionizing Radiation Gauging Devices, Nat. Bur. Stand. (U.S.), Handb. 129, 29 pages (Oct. 1979) Stock No. 003-003-02135-1, $1.75.

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Hockey, B. J., and Rice, R. W., Eds., The Science of Ceramic Machining and Surface Finishing II. Proceedings of a Symposium held at the National Bureau of Standards, Gaithersburg, MD, Nov. 13-15, 1978, Nat. Bur. Stand. (U.S.), Spec. Publ. 562, 524 pages (Oct. 1979) Stock No. 003-003-02122-9, $9.00.

Properties of Materials: Electronic, Magnetic, and Optical

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Morris, M. C., McMurdie, H. F., Evans, E. H., Paretzkin, B., and de Groot, J. H., Standard X-Ray Diffraction Powder Patterns, Nat. Bur. Stand. (U.S.), Monogr. 25-Sec. 16, 190 pages (Oct. 1979) Stock No. 003-003-02128-8, $5.00.

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Technology Incentives

Bell, J., Kirby, S., Weiss, R. G., Watson, S., The FCC Public Message Services Policy Change: An ETIP Evaluability Assessment Report—Volume 1, Nat. Bur. Stand. (U.S.), Tech. Note 1104/1, 232 pages (Sept. 1979) Stock No. 003-003-02117-2, $11.00. Sold in sets only.

Bell, J., Kirby, S., Weiss, R. G., and Watson, S., The FCC Public Message Services Policy Change: An ETIP Evaluability Assessment Report—Volume 2, Nat. Bur. Stand. (U.S.), Tech. Note 1104/2, 110 pages (Sept. 1979) Stock No. 003-003-02117-2, $11.00. Sold in sets only.

NEWS BRIEFS

CONGRESSIONAL HEARINGS ON NBS AUTHORIZATIONS. Science and technology trends of the 1980's and the role of NBS in dealing with these issues were discussed by NBS Director Ernest Ambler during a series of Congressional hearings on the proposed FY 1981 NBS budget and authorization bill. Conducted by the House Subcommittee on Science, Research and Technology, the hearings reviewed ongoing programs, new priorities, and plans for the future. Assistant Secretary of Commerce for Science and Technology Jordan Baruch and officials from each of the Bureau's major research groups also testified on NBS activities.

RAILWAY SCALE CALIBRATION WORK TRANSFERRED. NBS has transferred its work in the calibration of railway master scales to the Federal Grain Inspection Service (FGIS) of the Department of Agriculture. The move was made after the passage of the Grain Standards Act gave the FGIS regulatory authority over the 36 railroad track scales used to measure shipments of grain for export. FGIS will continue the NBS calibration program in cooperation with NBS, the Association of American Railroads, and the State of Illinois, which will take over the operation of the Bureau's railroad scale calibration center in Clearing, Illinois.

STANDARD REFERENCE DATA GRANTS OFFERED. The NBS Office of Standard Reference Data is managing a new interagency grants program for the critical evaluation of scientific data. Together with the National Science Foundation, the Department of Energy, and the Office of Naval Research, the OSRD will award grants for projects to review, evaluate, and compile published research data of interest to scientists and engineers. Projects are expected to be limited to specific, well-defined areas (such as rate constants for a class of chemical reactions) and to emphasize the critical evaluation of the data. Details are available from the OSRD, NBS, Washington, D.C. 20234, telephone 301/921-2467.

NEW PROBE FOR MEASURING ELECTROMAGNETIC RADIATION. A broadband electric-field sensor to measure the continuous-wave electromagnetic environment has been developed by NBS. The device offers a unique combination of frequency range, sensitivity range, and isotropic characteristics (independence of position in the field). The typical tangential sensitivity of the sensors is 13 to 16 microvolts per meter with usable dynamic range of 125 to 144 decibels for various bandwiths in the frequency range of 10 megahertz to 1 gigahertz. NBS developed the probe for the Environmental Protection Agency but NBS will provide circuit details to interested manufacturers.

SRM FOR USE IN ASSESSING STRAY LIGHT. NBS announces the availability of SRM 2032, Crystalline Potassium Iodide, for use in the assessment of radiant energy (stray light) in ultraviolet absorption spectrophotometers below 260 nm. Specific absorbance of an aqueous solution of SRM 2032 is certified at eight wavelengths in 5-nm intervals from 240 to 275 nm. Stray light in an ultraviolet spectrophotometer is determined by comparing the certified values of specific absorbance to the absorbance values of aqueous solutions of SRM 2032 measured under conditions of known concentration, pathlength, and temperature. SRM 2032 may be purchased from the Office of Standard Reference Materials, B311 Chemistry Building, NBS, Washington, D.C. 20234, for $152.

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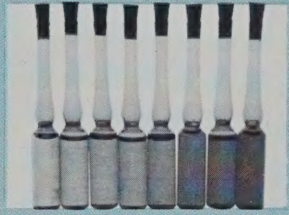

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